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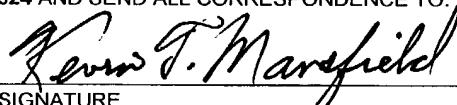
Form PTO-1390 (REV 10-95)		U S Department of Commerce Patent and Trademark Office	ATTORNEY'S DOCKET NUMBER <b>HF/5-22104/A/PCT</b>
<b>TRANSMITTAL LETTER TO THE UNITED STATES DESIGNATED/ELECTED OFFICE (DO/EO/US) CONCERNING A FILING UNDER 35 U.S.C. 371</b>		U.S. APPLICATION NO. (If known, see 37 CFR 1.5)	
INTERNATIONAL APPLICATION NO. <b>PCT/EP 00/09394</b>	INTERNATIONAL FILING DATE <b>September 26, 2000</b>	<b>10/089851</b>	
TITLE OF INVENTION <b>FABRIC SOFTENER COMPOSITIONS</b>		PRIORITY DATE CLAIMED <b>October 5, 1999</b>	
APPLICANT(S) FOR DO/EO/US <b>Petr Kvita, Peter Otto, Mario Dubini, Harald Chrobaczek, Michael Geubtner, Ralf Goretzki, Barbara Weber and Emmanuel Martin</b>			

Applicant herewith submits to the United States Designated/Elected Office (DO/EO/US) the following items and other information:

1.  This is a **FIRST** submission of items concerning a filing under 35 U.S.C. 371.
2.  This is a **SECOND** or **SUBSEQUENT** submission of items concerning a filing under 35 U.S.C. 371.
3.  This express request to begin national examination procedures (35 U.S.C. 371(f) at any time rather than delay examination until the expiration of the applicable time limit set in 35 U.S.C. 371(b) and PCT Articles 22 and 39 (1).
4.  A proper Demand for International Preliminary Examination was made by the 19th month from the earliest claimed priority date.
5.  A copy of the International Application as filed (35 U.S.C. 371(c)(2))
  - a.  is transmitted herewith (required only if not transmitted by the International Bureau).
  - b.  has been transmitted by the International Bureau. (See attached Form PCT/IB/308)
  - c.  is not required, as the application was filed in the United States Receiving Office (RO/US).
6.  A translation of the International Application into English 35.U.S.C. 371(c)(2)).
7.  Amendments to the claims of the International Application under PCT Article 19 (35 U.S.C.371(c)(3)).
  - a.  are transmitted herewith (required only if not transmitted by the International Bureau).
  - b.  have been transmitted by the International Bureau.
  - c.  have not been made; however, the time limit for making such amendments has NOT expired.
  - d.  have not been made and will not be made.
8.  A translation of the amendments to the claims under PCT Article 19 (35 U.S.C. 371 (c)(3)).
9.  An oath or declaration of the inventor(s) (35 U.S.C. 371(c)(4)).
10.  A translation of the annexes to the International Preliminary Examination Report under PCT Article 36 (35 U.S.C. 371(c)(5)).

**Items 11. to 16. below concern document(s) or information included.**

11.  An Information Disclosure Statement under 37 CFR 1.97 and 1.98.
12.  An assignment document for recording. A separate cover sheet in compliance with 37 CFR 3.28 and 3.31 is included.
13.  **A FIRST preliminary amendment.**
 A SECOND or SUBSEQUENT preliminary amendment.
14.  A substitute specification.
15.  A change of power of attorney and/or address letter.
16.  Other items or information: (See attached Form PCT/ISA/210)

U.S. APPLICATION NO. (if known, see 37 CFR 1.5)	INTERNATIONAL APPLICATION NO PCT/EP 00/09394	ATTORNEY'S DOCKET NUMBER HF/5-22104/A/PCT		
17. <input checked="" type="checkbox"/> The following fees are submitted: <b>BASIC NATIONAL FEE (37 CFR 1.492(a) (1)-(5)):</b>		<b>\$890.00</b>		
Search Report has been prepared by the EPO or JPO .....		\$890.00		
International preliminary examination fee paid to USPTO (37 CFR 1.482) .....		\$710.00		
No international preliminary examination fee paid to USPTO (37 CFR 1.482) but international search fee paid to USPTO (37 CFR 1.445(a)(2)).....		\$740.00		
Neither international preliminary examination fee (37 CFR 1.482) nor international search fee (37 CFR 1.445(a)(2)) paid to USPTO.....		\$1040.00		
International preliminary examination fee paid to USPTO (37 CFR 1.482) and all claims satisfied provisions of PCT Article 33(2)-(4). ....		\$100.00		
<b>ENTER APPROPRIATE BASIC FEE AMOUNT = \$890.00</b>				
Surcharge of <b>\$130.00</b> for furnishing the oath of declaration later than <input type="checkbox"/> 20 <input type="checkbox"/> 30 months from the earliest claimed priority date (37 CFR 1.492(e)).		\$		
CLAIMS	NUMBER FILED	NUMBER EXTRA	RATE	
Total claims	21 - 20 =	1	X \$18.00	\$18.00
Independent claims	1 - 3 =	0	X \$84.00	\$
MULTIPLE DEPENDENT CLAIM(S) (if applicable)			+ \$280.00	\$
			<b>TOTAL OF ABOVE CALCULATIONS =</b>	<b>\$908.00</b>
Reduction of 1/2 for filing by small entity, if applicable. Verified Small Entity Statement must also be filed (Note 37 CFR 1.9, 1.27, 1.28).				\$
			<b>SUBTOTAL =</b>	<b>\$908.00</b>
Processing fee of <b>\$130.00</b> for furnishing the English translation later than <input type="checkbox"/> 20 <input type="checkbox"/> 30 months from the earliest claimed priority date (37 CFR 1.492(f)).				\$
				+ \$
			<b>TOTAL NATIONAL FEE =</b>	<b>\$908.00</b>
Fee for recording the enclosed assignment (37 CFR 1.21(h)). The assignment must be accompanied by an appropriate cover sheet (37 CFR 3.28, 3.31). <b>\$40.00</b> per property				\$
			<b>TOTAL FEES ENCLOSED =</b>	<b>\$</b>
			Amount to be: refunded	\$
			charged	\$908.00
<p>a. <input checked="" type="checkbox"/> Please charge my Deposit Account No. 03-1935 in the amount of <b>\$908.00</b> to cover the above fees. A duplicate copy of this sheet is enclosed.</p> <p>b. <input checked="" type="checkbox"/> The Commissioner is hereby authorized to charge any additional fees which may be required, or credit any overpayment to Deposit Account No. 03-1935. A duplicate copy of this sheet is enclosed.</p>				
<p><b>NOTE: Where an appropriate time limit under 37 CFR 1.494 or 1.495 has not been met, a petition to revive (37 CFR 1.137(a) or (b)) must be filed and granted to restore the application to pending status.</b></p>				
<p>PLEASE ASSOCIATE THE ATTACHED APPLICATION WITH CUSTOMER NUMBER 000324 AND SEND ALL CORRESPONDENCE TO:</p>				
<p>JoAnn Villamizar, Ciba Specialty Chemicals Corporation Patent Department 540 White Plains Road P.O. Box 2005 Tarrytown, NY 10591-9005</p>				
<p> SIGNATURE</p>				
<p>Kevin T. Mansfield NAME Reg. No. 31,635</p>				
<p>APR 03 2002</p>				

CASE HF/5-22104/A/PCT

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

IN RE PCT NATIONAL STAGE APPLICATION OF  
PETR KVITA ET AL  
INTERNATIONAL APPLICATION NO. PCT/EP 00/09394  
FILED: SEPTEMBER 26, 2000  
FOR: FABRIC SOFTENER COMPOSITIONS  
U.S. APPLICATION NO: UNASSIGNED  
35 USC 371 DATE:

Group Art Unit:  
Examiner:

Assistant Commissioner for Patents  
Washington, D.C. 20231

**PRELIMINARY AMENDMENT**

Sir:

Kindly amend this application as follows prior to calculation of the filing fee and consideration on the merits.

**IN THE CLAIMS**

Please cancel claims 1-21.

Please add the following claims.

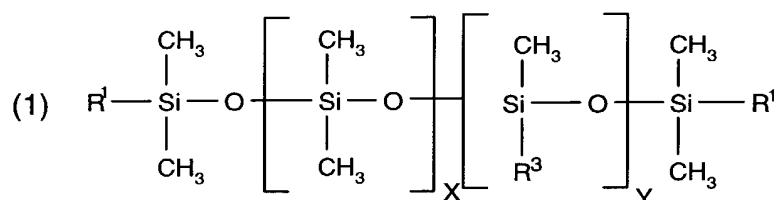
--22. (new) A method of use of a softener composition for the antipilling treatment of textile fibre materials in domestic applications, which comprises treating washed textile fibre materials with a softener composition which comprises:

A) a fabric softener;

B) at least one additive selected from the group consisting of

- a) a polyethylene, or a mixture thereof,
- b) a fatty acid alkanolamide, or a mixture thereof,
- c) a polysilicic acid, or a mixture thereof, and
- d) a polyurethane, or a mixture thereof; and

C) a dispersed polyorganosiloxane of formula (1)

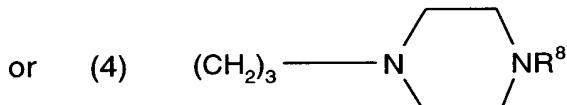
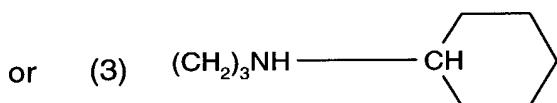
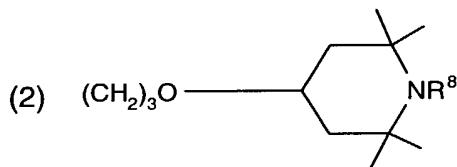


wherein

$\text{R}^1$  is OH, OR<sup>2</sup> or CH<sub>3</sub>,

$\text{R}^2$  is CH<sub>3</sub> or CH<sub>2</sub>CH<sub>3</sub>,

$\text{R}^3$  is C<sub>1</sub>-C<sub>20</sub>alkoxy, CH<sub>3</sub>, CH<sub>2</sub>CHR<sup>4</sup>CH<sub>2</sub>NHR<sup>5</sup>, or CH<sub>2</sub>CHR<sup>4</sup>CH<sub>2</sub>N(COCH<sub>3</sub>)R<sup>5</sup>,



$\text{R}^4$  is H or CH<sub>3</sub>,

$\text{R}^5$  is H, CH<sub>2</sub>CH<sub>2</sub>NHR<sup>6</sup>, C(=O)-R<sup>7</sup> or (CH<sub>2</sub>)<sub>z</sub>-CH<sub>3</sub>,

$z$  is 0 to 7,

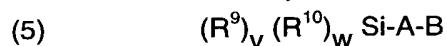
$\text{R}^6$  is H or C(=O)-R<sup>7</sup>,

$\text{R}^7$  is CH<sub>3</sub>, CH<sub>2</sub>CH<sub>3</sub> or CH<sub>2</sub>CH<sub>2</sub>CH<sub>2</sub>OH,

$\text{R}^8$  is H or CH<sub>3</sub>, and

the sum of X and Y is 40 to 4000;

or a dispersed polyorganosiloxane which comprises at least one unit of the formula (5)



wherein

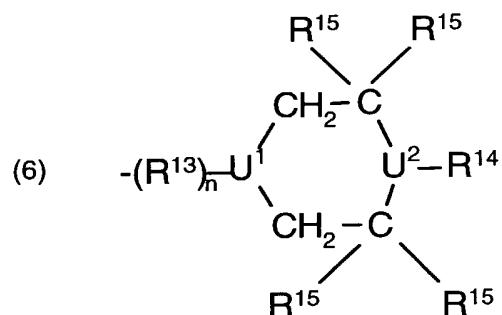
$R^9$  is  $CH_3$ ,  $CH_3CH_2$  or phenyl,

$R^{10}$  is  $-O-Si$  or  $-O-R^9$ ,

the sum of  $v$  and  $w$  equals 3, and  $v$  does not equal 3,

$A = -CH_2CH(R^{11})(CH_2)_k$ ,

$B = -NR^{12}((CH_2)_l-NH)_mR^{12}$  or



$n$  is 0 or 1,

when  $n$  is 0,  $U^1$  is N, when  $n$  is 1,  $U^1$  is CH,

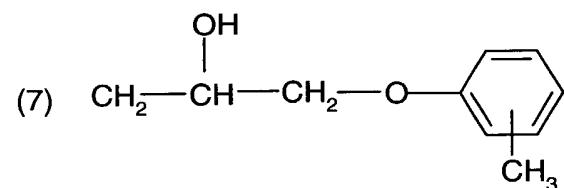
$l$  is 2 to 8,

$k$  is 0 to 6,

$m$  is 0 to 3,

$R^{11}$  is H or  $CH_3$ ,

$R^{12}$  is H,  $C(=O)-R^{16}$ ,  $CH_2(CH_2)_pCH_3$  or



$p$  is 0 to 6,

$R^{13}$  is NH, O,  $OCH_2CH(OH)CH_2N$ (butyl), or  $OOCN$ (butyl),

$R^{14}$  is H, linear or branched  $C_1-C_4$  alkyl, phenyl or  $CH_2CH(OH)CH_3$ ,

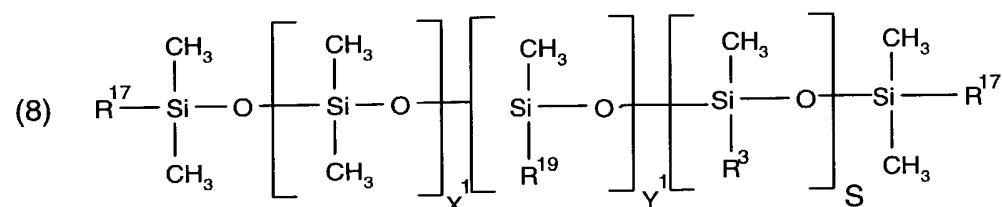
$R^{15}$  is H or linear or branched  $C_1-C_4$  alkyl,

$R^{16}$  is  $CH_3$ ,  $CH_2CH_3$  or  $(CH_2)_qOH$ ,

q is 1 to 6, and

$U^2$  is N or CH;

or a dispersed polyorganosiloxane of the formula (8)



wherein

$R^3$  is as previously defined,

$R^{17}$  is OH,  $OR^{18}$  or  $CH_3$ ,

$R^{18}$  is  $CH_3$  or  $CH_2CH_3$ ,

$R^{19}$  is  $R^{20}-(EO)_m-(PO)_n-R^{21}$ ,

m is 3 to 25,

n is 0 to 10,

$R^{20}$  is the direct bond or  $CH_2CH(R^{22})(CH_2)_pR^{23}$ ,

p is 1 to 4,

$R^{21}$  is H,  $R^{24}$ ,  $CH_2CH(R^{22})NH_2$  or  $CH(R^{22})CH_2NH_2$ ,

$R^{22}$  is H or  $CH_3$ ,

$R^{23}$  is O or NH,

$R^{24}$  is linear or branched  $C_1-C_8$  alkyl or  $Si(R^{25})_3$ ,

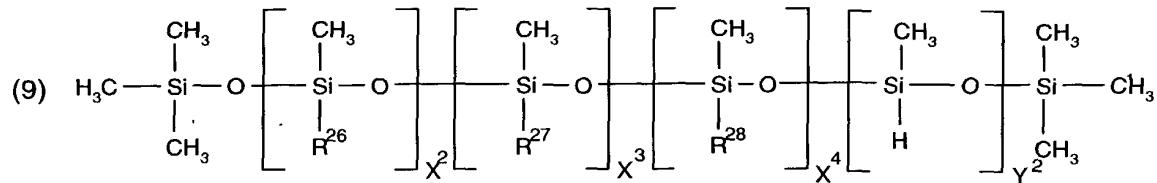
$R^{25}$  is  $R^{24}$ ,  $OCH_3$  or  $OCH_2CH_3$ ,

EO is  $-CH_2CH_2O-$ ,

PO is  $-CH(CH_3)CH_2O-$  or  $-CH_2CH(CH_3)O-$ , and

the sum of  $X_1, Y_1$  and S is 20 to 1500;

or a dispersed polyorganosiloxane of the formula (9)



wherein

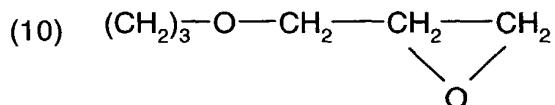
$R^{26}$  is linear or branched  $C_1$ - $C_{20}$ alkoxy,  $CH_2CH(R^4)R^{29}$ ,

$R^4$  is as previously defined,

$R^{29}$  is linear or branched  $C_1$ - $C_{20}$ alkyl,

$R^{27}$  is aryl, aryl substituted by linear or branched  $C_1$ - $C_{10}$ alkyl, linear or branched  $C_1$ - $C_{20}$ alkyl substituted by aryl or aryl substituted by linear or branched  $C_1$ - $C_{10}$ alkyl

$R^{28}$  is

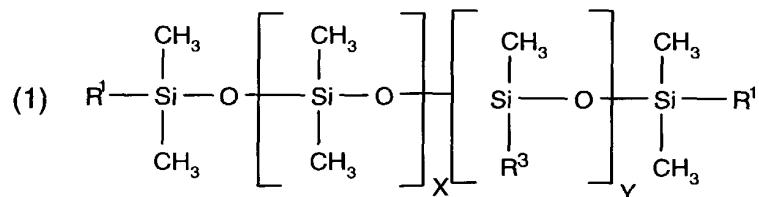


the sum of  $X^2$ ,  $X^3$ ,  $X^4$  and  $Y^2$  is 20 to 1500, wherein  $X^3$ ,  $X^4$  and  $Y^2$  may be independently of each other

0;

or a mixture thereof.

23. (new) A method of use according to claim 22 wherein the polyorganosiloxane is of formula (1):

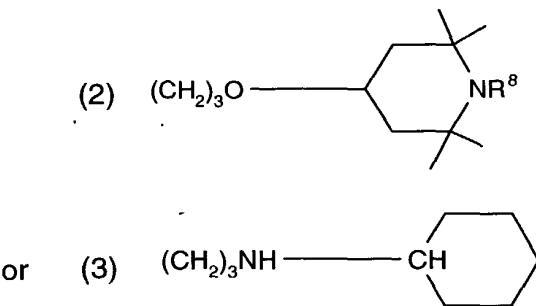


wherein

$R^1$  is  $OH$ ,  $OR^2$  or  $CH_3$ ,

$R^2$  is  $CH_3$  or  $CH_2CH_3$ ,

$R^3$  is  $C_1$ - $C_{20}$ alkoxy,  $CH_3$ ,  $CH_2CHR^4CH_2NHR^5$ , or



$R^4$  is H or  $CH_3$ ,

$R^5$  is H,  $CH_2CH_2NHR^6$ ,  $C(=O)-R^7$ ,

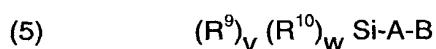
$R^6$  is H or  $C(=O)-R^7$ ,

$R^7$  is  $CH_3$ ,  $CH_2CH_3$  or  $CH_2CH_2CH_2OH$ ,

$R^8$  is H or  $CH_3$ , and

the sum of X and Y is 40 to 1500;

or a dispersed polyorganosiloxane which comprises at least one unit of the formula (5);



wherein

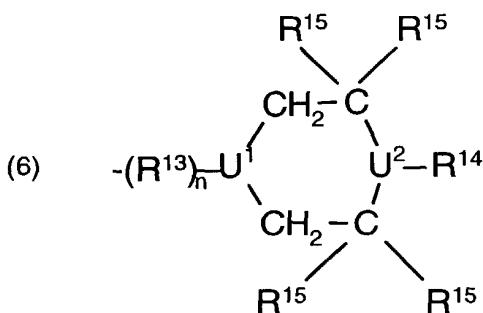
$R^9$  is  $CH_3$ ,  $CH_3CH_2$ ,

$R^{10}$  is  $-O-Si$  or  $-O-R^9$ ,

the sum of v and w equals 3, and v does not equal 3,

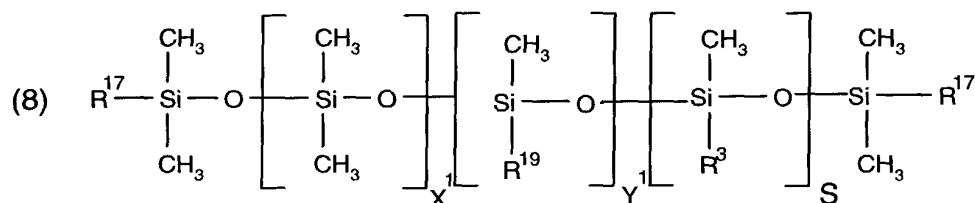
A =  $-CH_2CH(R^{11})(CH_2)_K$ ,

B =



n is 1,  
 U<sup>1</sup> is CH,  
 k is 0 to 6,  
 R<sup>11</sup> is H or CH<sub>3</sub>,  
 R<sup>13</sup> is OOCN(butyl),  
 R<sup>14</sup> is H, linear C<sub>1</sub>-C<sub>4</sub> alkyl, phenyl,  
 R<sup>15</sup> is H or linear C<sub>1</sub>-C<sub>4</sub>alkyl, and  
 U<sup>2</sup> is N;

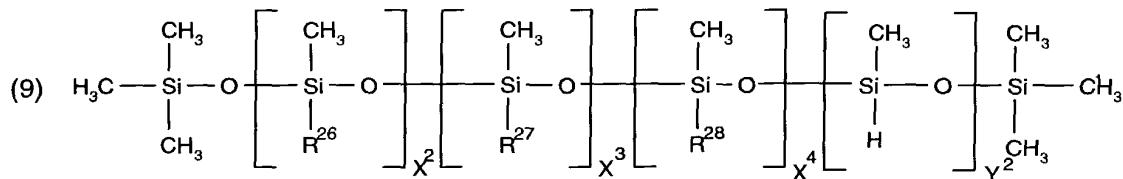
or a dispersed polyorganosiloxane of the formula (8);



wherein

R<sup>3</sup> is as previously defined,  
 R<sup>17</sup> is OH, OR<sup>18</sup> or CH<sub>3</sub>,  
 R<sup>18</sup> is CH<sub>3</sub> or CH<sub>2</sub>CH<sub>3</sub>,  
 R<sup>19</sup> is R<sup>20</sup>-(EO)<sub>m</sub>-(PO)<sub>n</sub>-R<sup>21</sup>,  
 m is 3 to 25,  
 n is 0 to 10,  
 R<sup>20</sup> is the direct bond or CH<sub>2</sub>CH(R<sup>22</sup>)(CH<sub>2</sub>)<sub>p</sub>R<sup>23</sup>,  
 p is 1 to 4,  
 R<sup>21</sup> is H, R<sup>24</sup>, CH<sub>2</sub>CH(R<sup>22</sup>)NH<sub>2</sub> or CH(R<sup>22</sup>)CH<sub>2</sub>NH<sub>2</sub>,  
 R<sup>22</sup> is H or CH<sub>3</sub>,  
 R<sup>23</sup> is O or NH,  
 R<sup>24</sup> is linear or branched C<sub>1</sub>-C<sub>3</sub> alkyl or Si(R<sup>25</sup>)<sub>3</sub>,  
 R<sup>25</sup> is R<sup>24</sup>, OCH<sub>3</sub> or OCH<sub>2</sub>CH<sub>3</sub>,  
 EO is -CH<sub>2</sub>CH<sub>2</sub>O-,  
 PO is -CH(CH<sub>3</sub>)CH<sub>2</sub>O- or -CH<sub>2</sub>CH(CH<sub>3</sub>)O-, and  
 the sum of X<sub>1</sub>, Y<sub>1</sub> and S is 40 to 1500;

or a dispersed polyorganosiloxane of the formula (9);



in which

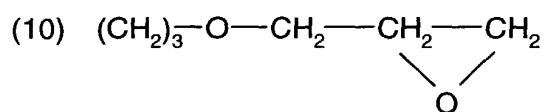
$\text{R}^{26}$  is linear  $\text{C}_1 - \text{C}_{20}$  alkoxy,

$\text{R}^4$  is as previously defined,

$\text{R}^{29}$  is linear  $\text{C}_1 - \text{C}_{20}$  alkyl,

$\text{R}^{27}$  is,  $\text{CH}_2\text{CH}(\text{R}^4)\text{phenyl}$  and

$\text{R}^{28}$  is



the sum of  $\text{X}^2$ ,  $\text{X}^3$ ,  $\text{X}^4$  and  $\text{Y}^2$  is 40 to 1500, wherein  $\text{X}^3$ ,  $\text{X}^4$  and  $\text{Y}^2$  may be independently of each other 0;

or a mixture thereof.

24. (new) A method of use according to claim 22 wherein a polyorganosiloxane of formula (1) is used, wherein

$\text{R}^1$  is  $\text{OH}$  or  $\text{CH}_3$ ,

$\text{R}^3$  is  $\text{CH}_3$ ,  $\text{C}_{10}-\text{C}_{20}$  alkoxy or  $\text{CH}_2\text{CHR}^4\text{CH}_2\text{NHR}^5$ ,

$\text{R}^4$  is  $\text{H}$ ,

$\text{R}^5$  is  $\text{H}$  or  $\text{CH}_2\text{CH}_2\text{NHR}^6$ ,

$\text{R}^6$  is  $\text{H}$  or  $\text{C}(\text{=O})-\text{R}^7$ , and

$\text{R}^7$  is  $\text{CH}_3$ ,  $\text{CH}_2\text{CH}_3$  or  $\text{CH}_2\text{CH}_2\text{CH}_2\text{OH}$ .

25. (new) A method of use according to claim 22 wherein a polyorganosiloxane of formula (8) is used, wherein

$\text{R}^3$  is  $\text{CH}_3$ ,  $\text{C}_{10}-\text{C}_{20}$  alkoxy or  $\text{CH}_2\text{CHR}^4\text{CH}_2\text{NHR}^5$ ,

$\text{R}^4$  is  $\text{H}$ ,

$\text{R}^5$  is  $\text{H}$  or  $\text{CH}_2\text{CH}_2\text{NHR}^6$ ,

$R^6$  is H or  $C(=O)-R^7$ ,  
 $R^7$  is  $CH_2CH_3$ ,  $CH_2CH_2CH_2OH$  or  $CH_3$ , and  
 $R_{17}$  is  $CH_3$  or OH.

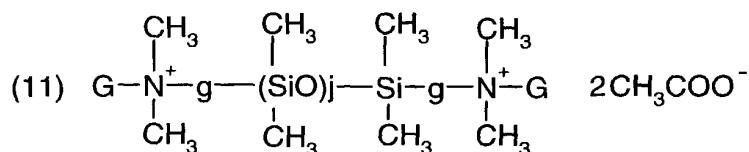
26. (new) A method of use according to claim 22 wherein a polyorganosiloxane of formula (9) is used, wherein

$R^{26}$  is  $CH_2CH(R^4)R^{29}$ ,

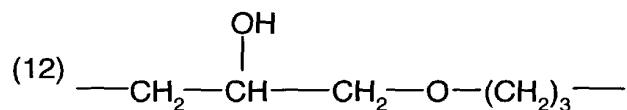
$R^4$  is H, and

$R^{27}$  is 2-phenyl propyl.

27. (new) A method of use according to claim 22 wherein the polyorganosiloxane composition comprises an additional polyorganosiloxane of the formula (11):



wherein g is



and G is  $C_1$  to  $C_{20}$  alkyl.

28. (new) A method of use according to claim 22 wherein the composition is a liquid aqueous composition.

29. (new) A method of use according to claim 22 wherein the composition is used in a tumble dryer sheet composition.

30. (new) A method of use according to claim 22 in which the polyorganosiloxane is nonionic or cationic.

31. (new) A method of use according to claim 22 in which the composition has a solids content of 5 to 70 % at a temperature of 120° C.

32. (new) A method of use according to claim 22 in which the composition contains a water content of 25 to 90 % by weight based on the total weight of the composition.

33. (new) A method of use according to claim 22 in which the composition has a pH value from 2 to 7.

34. (new) A method of use according to claim 22 in which the nitrogen content of the aqueous emulsion due to the polyorganosiloxane is from 0 to 0.25 % with respect to the silicon content.

35. (new) A method of use according to claim 22 wherein the composition comprises a polyethylene, a fatty acid alkanolamide or a polyurethane.

36. (new) A method of use according to claim 22 wherein the composition comprises a polyethylene or a fatty acid alkanolamide.

37. (new) A method of use according to claim 22 wherein the composition comprises a fatty acid alkanolamide.

38. (new) A method of use according to claim 22 wherein the composition comprises a polyethylene.

39. (new) A method of use according to claim 22 wherein the composition is prepared by mixing a preformulated fabric softener with an emulsion comprising the polyorganosiloxane and the additive.

40. (new) A method of use according to claim 22 wherein composition has a clear appearance.

41. (new) A method of use according to claim 22 in which the composition comprises:

- a) 0.01 to 70 % by weight, based on the total weight of the composition, of a polyorganosiloxane, or a mixture thereof;
- b) 0.2 to 25 % by weight based on the total weight of an emulsifier, or a mixture thereof;
- c) 0.01 to 15 % by weight based on the total weight of at least one additive selected from the group consisting of a polyethylene, a fatty acid alkanolamide, a polysilicic acid and a polyurethane, and
- d) water to 100 %.

42 (new) A tumble dryer sheet comprising a composition as defined in claim 22.--

REMARKS

Claims 22-42 are pending. Claims 1-21 have been replaced by added claims 22-42. Claims 3-20 were replaced to correct informalities and reduce filing fees by eliminating multiple dependency. Claim 1 was replaced to provide minor clarification. Claim 22 was replaced to change dependency.

Newly added claims 22-42 are supported by originally filed claims 1-21 and the disclosure at page 6, third full paragraph. No new matter has been added.

Applicants aver that the claims are now in proper form for examination. An Action on the merits of the claims is respectfully awaited.

Respectfully submitted,



Kevin T. Mansfield  
Agent for Applicants  
Reg. No. 31,635

Ciba Specialty Chemicals Corporation  
Patent Department  
540 White Plains Road  
P.O. Box 2005  
Tarrytown, NY 10591-9005  
(914) 785-7127  
KTM22104PA

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## FABRIC SOFTENER COMPOSITIONS

### FIELD OF THE INVENTION

The present invention relates to the use of fabric softener compositions comprising selected polyorganosiloxanes, or mixtures thereof, together with selected additives for the antipilling treatment of textile materials in domestic applications. In particular it relates to textile softening compositions for use in a textile laundering operation to impart excellent antipilling benefits on the textile.

### BACKGROUND OF THE INVENTION

As is well known, the pill formed on worn clothing markedly detracts from the appearance and feel of the clothing. The occurrence of pill is particularly a problem in the field of knitted materials, so that it has been greatly desired to seek measures for preventing the occurrence of pill on knitted fibre materials. Methods of improving the feel of worn clothing are known, such as rinse-added softener compositions. Typically, such compositions contain a water-insoluble quaternary-ammonium fabric softening agent. Silicones have also been used in rinse-cycle softening compositions for various reasons.

As given above one component of the compositions of the present invention are polyorganosiloxanes. Such compounds are known to be used on an industrial scale to finish fabrics by providing them with a permanent or semi-permanent finish aimed at improving their general appearance. Significant for these industrial fabric finishing processes is a so-called curing step generally involving temperatures in excess of 150°C often for periods of one hour or more. The object here is to form a chemical finish which resists destruction during subsequent cleaning/laundering of fabrics. This process of finishing is not carried out in domestic applications and accordingly one would not expect benefits of a comparable nature or magnitude from polyorganosiloxanes included as adjuncts in domestic softeners. Indeed, it is noteworthy that if the compounds of the current invention achieved a permanence associated with industrial textile finishing, problems associated with a cumulative build through the wash cycles could occur such as fabric discoloration and even in extremes an unpleasant feel to the wearer.

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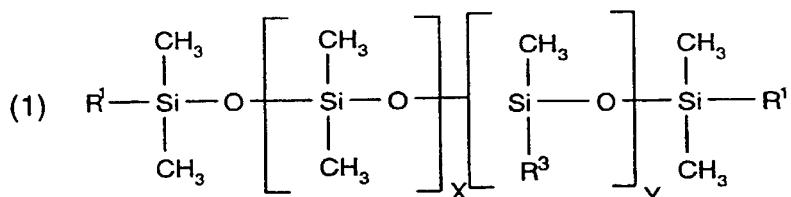
Surprisingly, it has been found that the use of selected polyorganosiloxanes, or mixtures thereof, together with selected additive in fabric softener compositions provide excellent antipilling effects when applied to fabrics during a textile laundry operation.

Similar benefits are noted when compositions of the current invention are incorporated into tumble dryer additives such as impregnates on sheets.

#### SUMMARY OF THE INVENTION

This invention relates to a method of use of a softener composition for the antipilling treatment of textile fibre materials in domestic applications, which softener composition comprises:

- A) a fabric softener;
- B) at least one additive selected from the group consisting of
  - a) a polyethylene, or a mixture thereof,
  - b) a fatty acid alkanolamide, or a mixture thereof,
  - c) a polysilicic acid, or a mixture thereof, and
  - d) a polyurethane, or a mixture thereof; and
- C) a dispersed polyorganosiloxane of formula (1)



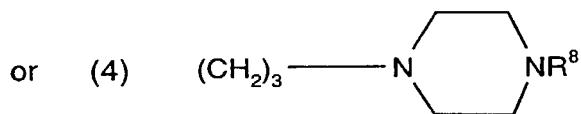
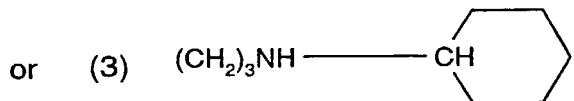
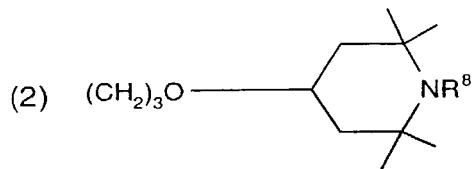
wherein

$\text{R}^1$  is  $\text{OH}$ ,  $\text{OR}^2$  or  $\text{CH}_3$

$\text{R}^2$  is  $\text{CH}_3$  or  $\text{CH}_2\text{CH}_3$

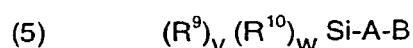
$\text{R}^3$  is  $\text{C}_{1-20}\text{alkoxy}$ ,  $\text{CH}_3$ ,  $\text{CH}_2\text{CHR}^4\text{CH}_2\text{NHR}^5$ , or  $\text{CH}_2\text{CHR}^4\text{CH}_2\text{N}(\text{COCH}_3)\text{R}^5$

- 3 -

 $\text{R}^4$  is H or  $\text{CH}_3$  $\text{R}^5$  is H,  $\text{CH}_2\text{CH}_2\text{NHR}^6$ ,  $\text{C}(=\text{O})-\text{R}^7$  or  $(\text{CH}_2)_z-\text{CH}_3$  $z$  is 0 to 7 $\text{R}^6$  is H or  $\text{C}(=\text{O})-\text{R}^7$  $\text{R}^7$  is  $\text{CH}_3$ ,  $\text{CH}_2\text{CH}_3$  or  $\text{CH}_2\text{CH}_2\text{CH}_2\text{OH}$  $\text{R}^8$  is H or  $\text{CH}_3$ 

the sum of X and Y is 40 to 4000;

or a dispersed polyorganosiloxane which comprises at least one unit of the formula (5)



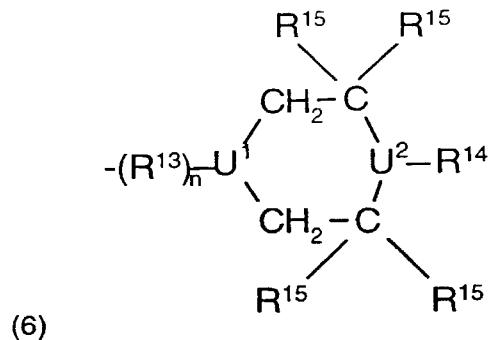
wherein

 $\text{R}^9$  is  $\text{CH}_3$ ,  $\text{CH}_3\text{CH}_2$  or Phenyl $\text{R}^{10}$  is  $-\text{O-Si}$  or  $-\text{O-R}^9$ 

the sum of v and w equals 3, and v does not equal 3

A =  $-\text{CH}_2\text{CH}(\text{R}^{11})(\text{CH}_2)_K$ B =  $-\text{NR}^{12}((\text{CH}_2)_l\text{-NH})_m\text{R}^{12}$ , or

- 4 -



n is 0 or 1

when  $n$  is 0,  $U^1$  is  $N$ , when  $n$  is 1,  $U^1$  is  $CH$

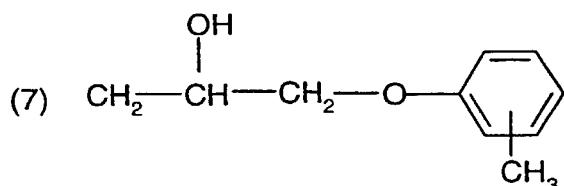
1 is 2 to 8

k is 0 to 6

m is 0 to 3

R<sup>11</sup> is H or CH<sub>3</sub>

$R^{12}$  is H,  $C(=O)-R^{16}$ ,  $CH_2(CH_2)_6CH_3$  or



p is 0 to 6

$R^{13}$  is NH, O,  $OCH_2CH(OH)CH_2N(Butyl)$ ,  $OOCN(Butyl)$

$R^{14}$  is H, linear or branched C<sub>1</sub>-C<sub>4</sub> alkyl, Phenyl or CH<sub>2</sub>CH(OH)CH<sub>3</sub>

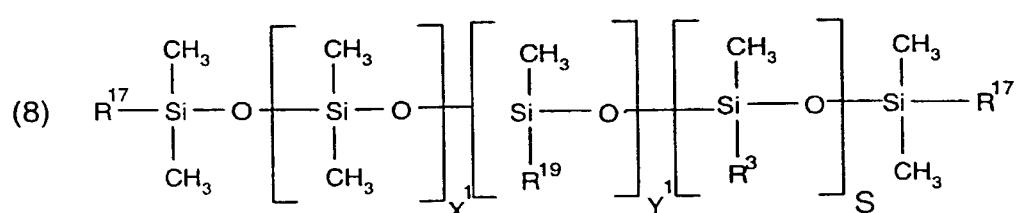
$B^{15}$  is H or linear or branched  $C_1$ - $C_4$  alkyl

$R^{16}$  is  $CH_3$ ,  $CH_2CH_3$  or  $(CH_2)_nOH$

g is 1 to 6

$\text{U}^2$  is N or CH.

or a dispersed polyorganosiloxane of the formula (8)



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wherein

$R^3$  is as previously defined

$R^{17}$  is OH, OR<sup>18</sup> or CH<sub>3</sub>

$R^{18}$  is CH<sub>3</sub> or CH<sub>2</sub>CH<sub>3</sub>

$R^{19}$  is  $R^{20}$ -(EO)<sub>m</sub>-(PO)<sub>n</sub>-R<sup>21</sup>

$m$  is 3 to 25

$n$  is 0 to 10

$R^{20}$  is the direct bond or CH<sub>2</sub>CH(R<sup>22</sup>)(CH<sub>2</sub>)<sub>p</sub>R<sup>23</sup>

$p$  is 1 to 4

$R^{21}$  is H, R<sup>24</sup>, CH<sub>2</sub>CH(R<sup>22</sup>)NH<sub>2</sub> or CH(R<sup>22</sup>)CH<sub>2</sub>NH<sub>2</sub>

$R^{22}$  is H or CH<sub>3</sub>

$R^{23}$  is O or NH

$R^{24}$  is linear or branched C<sub>1</sub>-C<sub>8</sub> alkyl or Si(R<sup>25</sup>)<sub>3</sub>

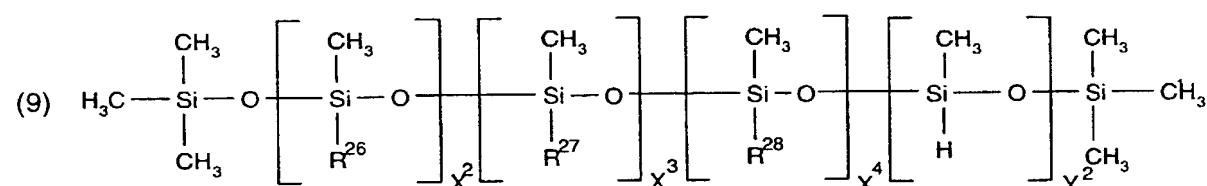
$R^{25}$  is R<sup>24</sup>, OCH<sub>3</sub> or OCH<sub>2</sub>CH<sub>3</sub>

EO is -CH<sub>2</sub>CH<sub>2</sub>O-

PO is -CH(CH<sub>3</sub>)CH<sub>2</sub>O- or -CH<sub>2</sub>CH(CH<sub>3</sub>)O-

the sum of X<sub>1</sub>, Y<sub>1</sub>, and S is 20 to 1500;

or a dispersed polyorganosiloxane of the formula (9)



wherein

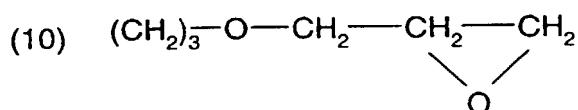
$R^{26}$  is linear or branched C<sub>1</sub> - C<sub>20</sub> alkoxy, CH<sub>2</sub>CH(R<sup>4</sup>)R<sup>29</sup>

$R^4$  is as previously defined

$R^{29}$  is linear or branched C<sub>1</sub> - C<sub>20</sub> alkyl

$R^{27}$  is aryl, aryl substituted by linear or branched C<sub>1</sub> - C<sub>10</sub> alkyl, linear or branched C<sub>1</sub> - C<sub>20</sub> alkyl substituted by aryl or aryl substituted by linear or branched C<sub>1</sub> - C<sub>10</sub> alkyl

$R^{28}$  is



- 6 -

the sum of  $X^2$ ,  $X^3$ ,  $X^4$  and  $Y^2$  is 20 to 1500, wherein  $X^3$ ,  $X^4$  and  $Y^2$  may be independently of each other 0;  
or a mixture thereof.

The composition is preferably used as a component in a liquid rinse conditioner composition.  
The textile fibre materials are treated for antipilling.

In tumble dryer applications the compositions are usually incorporated into impregnates on non-woven sheets. However, other application forms are known to those skilled in the art.

The fabric softener composition will be used after the textile fibre materials have been washed with a laundry detergent, which may be one of a broad range of detergent types. The tumble dryer sheet will be used after a laundering process. The textile fibre materials may be damp or dry.

The fabric softener composition may also be sprayed directly onto the fabrics prior to or during the ironing or drying of the treated fabrics.

The polyorganosiloxane may be anionic, nonionic or cationic, preferably nonionic or cationic.

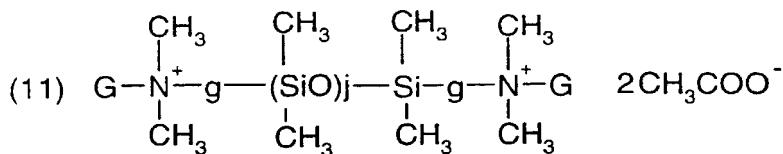
The polyorganosiloxanes, or mixtures thereof, are usually used in a dispersed form, via the use of an emulsifier. The fabric softener composition preferably contains a water content of 25 to 90% by weight based on the total weight of the emulsion.

When the polyorganosiloxane contains a nitrogen atom, the nitrogen content of the aqueous emulsion due to the polyorganosiloxane is as a rule from 0.001 to 0.25 % with respect to the silicon content. In general, a nitrogen content from 0 to 0.25 % is preferred. The particles of the emulsion usually have a diameter of between 5nm and 1000nm.

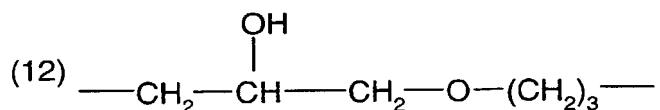
The fabric softener composition preferably has a solids content of 5 to 70% at a temperature of 120°C.

The fabric softener composition preferably has a pH value from 2 to 9.0, especially 2 to 7.

The fabric softener composition may further comprise an additional polyorganosiloxane:



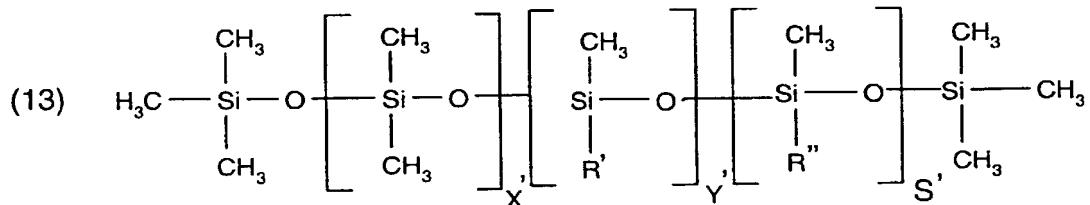
wherein g is



and G is C<sub>1</sub> to C<sub>20</sub> alkyl.

This polydimethylsiloxane is cationic, has a viscosity at 25°C of 250 mm<sup>2</sup>s<sup>-1</sup> to 450 mm<sup>2</sup>s<sup>-1</sup>, has a specific gravity of 1.00 to 1.02 g/cm<sup>3</sup> and has a surface tension of 28.5 mNm<sup>-1</sup> to 33.5 mNm<sup>-1</sup>.

The fabric softener composition may further comprise an additional polyorganosiloxane, such as that known as Magnasoft HSSD, or a polyorganosiloxane of the formula:



R''' is CH<sub>2</sub>CH<sub>2</sub>CH<sub>2</sub>N(R'')<sub>2</sub>

R'' is linear or branched C<sub>1</sub>-C<sub>4</sub> alkyl

R' is (CH<sub>2</sub>)<sub>x</sub>-(EO)<sub>m</sub>-(PO)<sub>n</sub>-R'''

m is 3 to 25

n is 0 to 10

X'' is 0 to 4

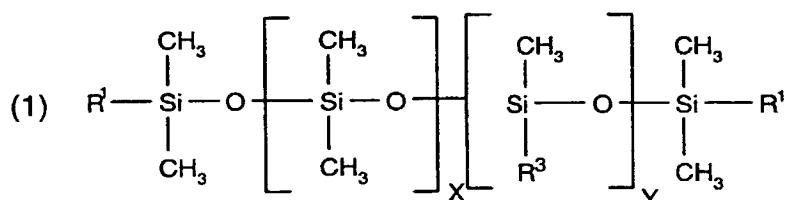
R''' is H or linear or branched C<sub>1</sub>-C<sub>4</sub> alkyl

EO is -CH<sub>2</sub>CH<sub>2</sub>O-

PO is -CH(CH<sub>3</sub>)CH<sub>2</sub>O- or -CH<sub>2</sub>CH(CH<sub>3</sub>)O-

the sum of X', Y and S is 40 to 300.

Preferably the compositions comprise dispersed polyorganosiloxanes of formula (1):

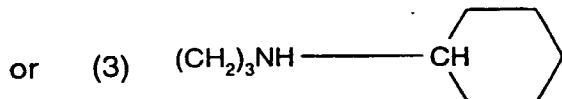
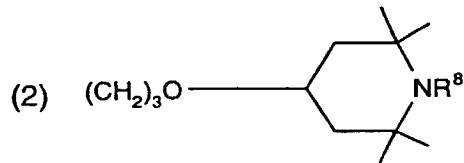


wherein

R<sup>1</sup> is OH, OR<sup>2</sup> or CH<sub>3</sub>

R<sup>2</sup> is CH<sub>3</sub> or CH<sub>2</sub>CH<sub>3</sub>

R<sup>3</sup> is C<sub>1</sub>-C<sub>20</sub>alkoxy, CH<sub>3</sub>, CH<sub>2</sub>CHR<sup>4</sup>CH<sub>2</sub>NHR<sup>5</sup>, or



R<sup>4</sup> is H or CH<sub>3</sub>

R<sup>5</sup> is H, CH<sub>2</sub>CH<sub>2</sub>NHR<sup>6</sup>, C(=O)-R<sup>7</sup>

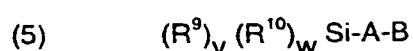
R<sup>6</sup> is H or C(=O)-R<sup>7</sup>

R<sup>7</sup> is CH<sub>3</sub>, CH<sub>2</sub>CH<sub>3</sub> or CH<sub>2</sub>CH<sub>2</sub>CH<sub>2</sub>OH

R<sup>8</sup> is H or CH<sub>3</sub>

the sum of X and Y is 40 to 1500

or a dispersed polyorganosiloxane which comprises at least one unit of the formula (5):



wherein

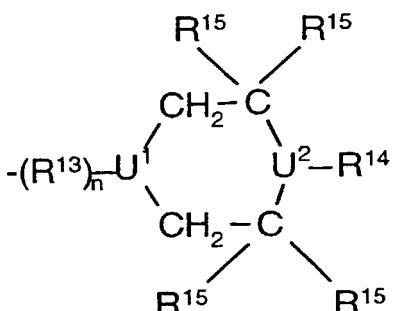
$R^9$  is  $CH_3$ ,  $CH_3CH_2$

$R^{10}$  is  $-O-Si$  or  $-O-R^9$

the sum of  $v$  and  $w$  equals 3, and  $v$  does not equal 3

$A = -CH_2CH(R^{11})(CH_2)_k$

$B =$



(6)

$n$  is 1

$U^1$  is CH

$k$  is 0 to 6

$R^{11}$  is H or  $CH_3$

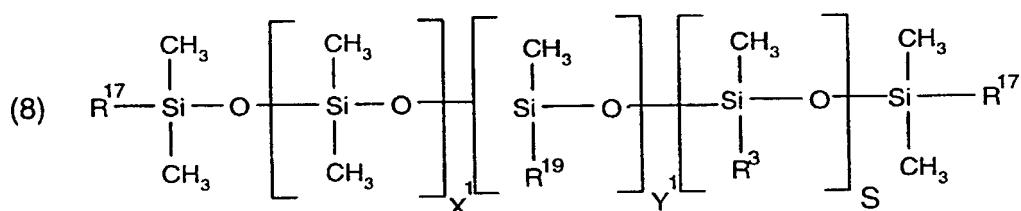
$R^{13}$  is  $OOCN(Butyl)$

$R^{14}$  is H, linear  $C_1-C_4$  alkyl, Phenyl

$R^{15}$  is H or linear  $C_1-C_4$  alkyl

$U^2$  is N

or a dispersed polyorganosiloxane of the formula (8);



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wherein

$R^3$  is as previously defined

$R^{17}$  is OH, OR<sup>18</sup> or CH<sub>3</sub>

$R^{18}$  is CH<sub>3</sub> or CH<sub>2</sub>CH<sub>3</sub>

$R^{19}$  is  $R^{20}$ -(EO)<sub>m</sub>-(PO)<sub>n</sub>-R<sup>21</sup>

$m$  is 3 to 25

$n$  is 0 to 10

$R^{20}$  is the direct bond or CH<sub>2</sub>CH(R<sup>22</sup>)(CH<sub>2</sub>)<sub>p</sub>R<sup>23</sup>

$p$  is 1 to 4

$R^{21}$  is H, R<sup>24</sup>, CH<sub>2</sub>CH(R<sup>22</sup>)NH<sub>2</sub> or CH(R<sup>22</sup>)CH<sub>2</sub>NH<sub>2</sub>

$R^{22}$  is H or CH<sub>3</sub>

$R^{23}$  is O or NH

$R^{24}$  is linear or branched C<sub>1</sub>-C<sub>3</sub> alkyl or Si(R<sup>25</sup>)<sub>3</sub>

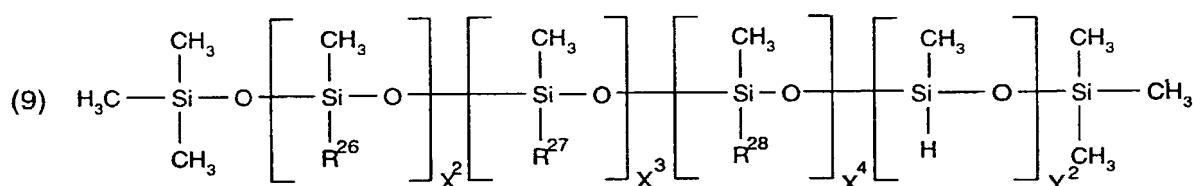
$R^{25}$  is R<sup>24</sup>, OCH<sub>3</sub> or OCH<sub>2</sub>CH<sub>3</sub>

EO is -CH<sub>2</sub>CH<sub>2</sub>O-

PO is -CH(CH<sub>3</sub>)CH<sub>2</sub>O- or -CH<sub>2</sub>CH(CH<sub>3</sub>)O-

the sum of X<sup>1</sup>, Y<sup>1</sup> and s is 40 to 1500

or a dispersed polyorganosiloxane of the formula (9);



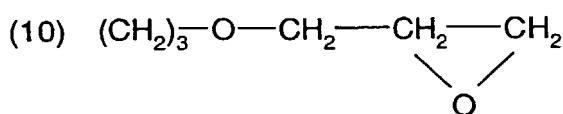
$R^{26}$  is linear C<sub>1</sub> - C<sub>20</sub> alkoxy,

$R^4$  is as previously defined

$R^{29}$  is linear C<sub>1</sub> - C<sub>20</sub> alkyl

$R^{27}$  is, CH<sub>2</sub>CH(R<sup>4</sup>)Phenyl

$R^{28}$  is



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the sum of  $X^2$ ,  $X^3$ ,  $X^4$  and  $Y^2$  is 40 to 1500, wherein  $X^3$ ,  $X^4$  and  $Y^2$  may be independently of each other 0; or a mixture thereof.

As to the polyorganosiloxanes of formula (1) the following preferences apply:

$R^1$  is preferably OH or  $CH_3$ .

$R^3$  is preferably  $CH_3$ ,  $C_{10-20}$ alkoxy or  $CH_2CHR^4CH_2NHR^5$ .

$R^4$  is preferably H.

$R^5$  is preferably H or  $CH_2CH_2NHR^6$ .

$R^6$  is preferably H or  $C(=O)-R^7$ .

$R^7$  is preferably  $CH_3$ ,  $CH_2CH_3$  or especially  $CH_2CH_2CH_2OH$ .

The sum of  $X + Y$  is preferably 100 to 2000.

Preferred are polyorganosiloxanes of formula (1) wherein

$R^1$  is OH or  $CH_3$ ,

$R^3$  is  $CH_3$ ,  $C_{10-20}$ alkoxy or  $CH_2CHR^4CH_2NHR^5$ ,

$R^4$  is H,

$R^5$  is H or  $CH_2CH_2NHR^6$ ,

$R^6$  is H or  $C(=O)-R^7$ , and

$R^7$  is  $CH_3$ ,  $CH_2CH_3$  or especially  $CH_2CH_2CH_2OH$ .

As to the polyorganosiloxanes of formula (8) the following preferences apply:

$R^3$  is preferably  $CH_3$ ,  $C_{10-20}$ alkoxy or  $CH_2CHR^4CH_2NHR^5$ .

$R^4$  is preferably H.

$R^5$  is preferably H or  $CH_2CH_2NHR^6$ .

$R^6$  is preferably H or  $C(=O)-R^7$ .

$R^7$  is preferably  $CH_2CH_3$ ,  $CH_2CH_2CH_2OH$  or especially  $CH_3$ .

$R_{17}$  is preferably  $CH_3$  or OH.

$R_{20}$  is preferably the direct bond.

$R_{21}$  is preferably H.

Preferred are polyorganosiloxanes of formula (8) wherein

$R^3$  is  $CH_3$ ,  $C_{10-20}$ alkoxy or  $CH_2CHR^4CH_2NHR^5$ ,

$R^4$  is H,

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R<sup>5</sup> is H or CH<sub>2</sub>CH<sub>2</sub>NHR<sup>6</sup>,

R<sup>6</sup> is H or C(=O)-R<sup>7</sup>,

R<sup>7</sup> is CH<sub>2</sub>CH<sub>3</sub>, CH<sub>2</sub>CH<sub>2</sub>CH<sub>2</sub>OH or especially CH<sub>3</sub>, and

R<sub>17</sub> is CH<sub>3</sub> or OH.

As to the polyorganosiloxanes of formula (9) the following preferences apply:

R<sup>26</sup> is preferably CH<sub>2</sub>CH(R<sup>4</sup>)R<sup>29</sup>.

R<sup>4</sup> is preferably H.

R<sup>27</sup> is preferably 2-phenyl propyl.

The sum of X<sup>2</sup>, X<sup>3</sup>, X<sup>4</sup> and Y<sup>2</sup> is preferably 40 to 500.

Preferred are polyorganosiloxanes of formula (9) wherein

R<sup>26</sup> is CH<sub>2</sub>CH(R<sup>4</sup>)R<sup>29</sup>,

R<sup>4</sup> is H, and

R<sup>27</sup> is 2-phenyl propyl.

Preferred are polyorganosiloxanes of formulae (1), (8) and (9), especially those of formulae (1) and (8). Very interesting polyorganosiloxanes are those of formula (1).

Emulsifiers used to prepare the polyorganosiloxane compositions include:

- i) Ethoxylates, such as alkyl ethoxylates, amine ethoxylates or ethoxylated alkylammoniumhalides. Alkyl ethoxylates include alcohol ethoxylates or isotridecyl ethoxylates. Preferred alcohol ethoxylates include linear or branched nonionic alkyl ethoxylates containing 2 to 15 ethylene oxide units. Preferred isotridecyl ethoxylates include nonionic isotridecyl ethoxylates containing 5 to 25 ethylene oxide units. Preferred amine ethoxylates include nonionic C10 to C20 alkyl amino ethoxylates containing 4 to 10 ethylene oxide units. Preferred ethoxylated alkylammoniumhalides include nonionic or cationic ethoxylated C6 to C20 alkyl bis(hydroxyethyl)methylammonium chlorides.
- ii) Alkylammonium halides, preferably cationic quaternary ester alkylammonium halides.
- iii) Silicones, preferably nonionic polydimethylsiloxane polyoxyalkylene copolymers
- iv) Saccharides, preferably nonionic alkylpolyglycosides.

A mixture of these emulsifiers may also be used.

As mentioned previously, the compositions further comprise one or more additives selected from polyethylene, dispersed fatty acid alkanol amide, polysilicic acid and polyurethane. These components are described below.

The emulsifiable polyethylene (polyethylene wax) is known and is described in detail in the prior art (compare, for example, DE-C-2,359,966, DE-A-2,824,716 and DE-A-1,925,993). The emulsifiable polyethylene is as a rule a polyethylene having functional groups, in particular COOH groups, some of which can be esterified. These functional groups are introduced by oxidation of the polyethylene. However, it is also possible to obtain the functionality by copolymerization of ethylene with, for example, acrylic acid. The emulsifiable polyethylenes have a density of at least 0.91 g/cm<sup>3</sup> at 20°C., an acid number of at least 5 and a saponification number of at least 10. Emulsifiable polyethylenes which have a density of 0.95 to 1.05 g/cm<sup>3</sup> at 20°C, an acid number of 10 to 60 and a saponification number of 15 to 80 are particularly preferred. Polyethylenes which have a drop point of 100-150°C are preferred. This material is generally obtainable commercially in the form of flakes, lozenges and the like. A mixture of these emulsifiable polyethylenes may also be used.

The polyethylene wax is employed in the form of dispersions. Various emulsifiers are suitable for their preparation. The preparation of the dispersions is described in detail in the prior art.

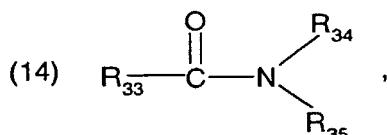
Emulsifiers suitable for dispersing the polyethylene component include:

- i) Ethoxylates, such as alkyl ethoxylates or amine ethoxylates. Alkyl ethoxylates include alcohol ethoxylates or isotridecyl ethoxylates. Preferred alcohol ethoxylates include nonionic fatty alcohol ethoxylates containing 2 to 55 ethylene oxide units. Preferred isotridecyl ethoxylates include nonionic isotridecyl ethoxylates containing 6 to 9 ethylene oxide units. Preferred amine ethoxylates include nonionic C10 to C20 alkyl amino ethoxylates containing 7 to 9 ethylene oxide units.
- ii) Alkylammonium halides, preferably cationic quaternary ester alkylammonium halides.
- iii) Ammonium salts, preferably cationic aliphatic quaternary ammonium chloride or sulfate.

A mixture of these emulsifiers may also be used.

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Suitable fatty acid alkanolamides are for example those of formula



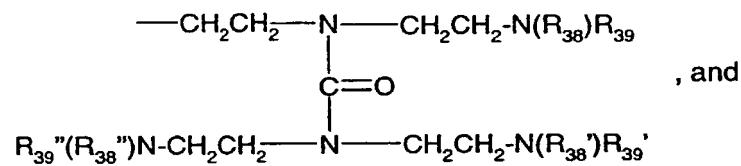
wherein

$\text{R}_{33}$  is a saturated or unsaturated hydrocarbon radical containing 10 to 24 carbon atoms,

$\text{R}_{34}$  is hydrogen or a radical of formula  $-\text{CH}_2\text{OH}$ ,  $-(\text{CH}_2\text{CH}_2\text{O})_c\text{H}$  or  $\begin{array}{c} \text{O} \\ \parallel \\ \text{---C---R}_{36} \end{array}$  wherein  $c$  is a

number from 1 to 10 and  $\text{R}_{36}$  is as defined above for  $\text{R}_{33}$ , and

$\text{R}_{35}$  is a radical of formula  $-\text{CH}_2\text{OH}$ ,  $-(\text{CH}_2\text{CH}_2\text{O})_c\text{H}$ ,  $\begin{array}{c} (\text{CH}_2\text{CH}_2\text{O})_c\text{H} \\ \diagup \\ \text{---CH}_2\text{CH}_2-\text{N} \end{array}$  or  $\begin{array}{c} \diagdown \\ \text{R}_{37} \end{array}$

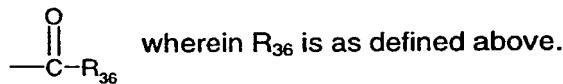


$\text{c}$  is as defined above,

$\text{R}_{37}$  is hydrogen or a radical of formula  $\begin{array}{c} \text{O} \\ \parallel \\ \text{---C---R}_{36} \end{array}$  wherein  $\text{R}_{36}$  is as defined above,

$\text{R}_{38}$ ,  $\text{R}_{38'}$  and  $\text{R}_{38''}$  have the same or different meaning and are as defined above for  $\text{R}_{34}$ , and

$\text{R}_{39}$ ,  $\text{R}_{39'}$  and  $\text{R}_{39''}$  have the same or different meaning and are a radical of formula

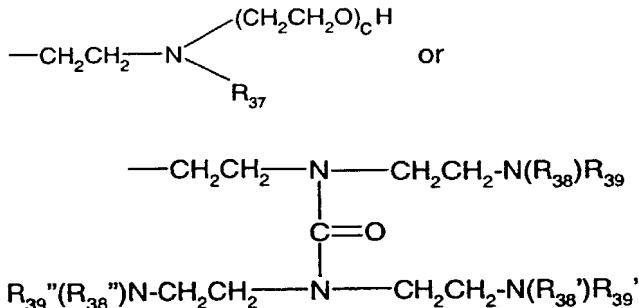


$\text{R}_{33}$  and  $\text{R}_{36}$  are preferably a saturated or unsaturated hydrocarbon radical containing 14 to 24 carbon atoms. Preferred are saturated hydrocarbon radicals.

$\text{R}_{34}$  is preferably hydrogen,  $-\text{CH}_2\text{OH}$  or a radical of formula  $\begin{array}{c} \text{O} \\ \parallel \\ \text{---C---R}_{36} \end{array}$ .

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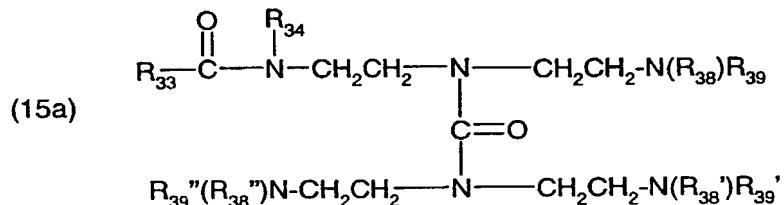
$R_{35}$  is preferably a radical of formula



As to  $R_{38}$ ,  $R_{38}'$  and  $R_{38}''$  the preferences given above for  $R_{34}$  apply.

$c$  is preferably a number from 1 to 5.

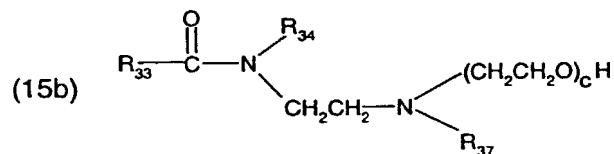
Preferred are fatty acid alkanolamides of formula



wherein  $R_{33}$ ,  $R_{34}$ ,  $R_{38}$ ,  $R_{38}'$ ,  $R_{38}''$ ,  $R_{39}$ ,  $R_{39}'$  and  $R_{39}''$  are as defined above.

Preferred are fatty acid alkanolamides of formula (15a), wherein  $R_{34}$ ,  $R_{38}$ ,  $R_{38}'$  and  $R_{38}''$  are hydrogen or  $-\text{CH}_2\text{OH}$ .

Furthermore, fatty acid alkanolamides of formula



are preferred, wherein  $R_{33}$ ,  $R_{34}$ ,  $R_{37}$  and  $c$  are as defined above.

Preferred are fatty acid alkanolamides of formula (15b), wherein

$R_{34}$  and  $R_{37}$  are hydrogen or a radical of formula  $\text{---C}(=\text{O})\text{---R}_{36}$ .  $R_{34}$  is preferably hydrogen.

The above fatty acid alkanolamides can also be present in form of the corresponding ammonium salts.

A mixture of these fatty acid alkanolamides may also be used.

Emulsifiers suitable for dispersing the fatty acid alkanol amide component include:

- i) Ethoxylates, such as alkyl ethoxylates, amine ethoxylates or amide ethoxylates. Alkyl ethoxylates include alcohol ethoxylates or isotridecyl ethoxylates. Preferred alcohol ethoxylates include nonionic fatty alcohol ethoxylates containing 2 to 55 ethylene oxide units. Preferred isotridecyl ethoxylates include nonionic isotridecyl ethoxylates containing 5 to 45 ethylene oxide units. Preferred amine ethoxylates include nonionic C10 to C20 alkyl amino ethoxylates containing 4 to 25 ethylene oxide units. Preferred amide ethoxylates include cationic fatty acid amide ethoxylates containing 2 to 25 ethylene oxide units.
- ii) Alkylammonium halides, preferably cationic quaternary ester alkylammonium halides or cationic aliphatic acid alkylamidotrialkylammonium methosulfates.
- iii) Ammonium salts, preferably cationic aliphatic quaternary ammonium chloride or sulfate.

A mixture of these emulsifiers may also be used.

Examples for polyurethanes are the reaction products of a diol and an ethoxysilane with a diisocyanate.

The additives selected from the group consisting of a polyethylene, a fatty acid alkanolamide, a polysilicic acid, and a polyurethane are, as a rule, used in an amount of 0.01 to 25 % by weight, especially 0.01 to 15 % by weight, based on the total weight of the fabric

softener composition. An amount of 0.05 to 15 % by weight, especially 0.1 to 15 % by weight, is preferred. Highly preferred is an upper limit of 10 %, especially 5 %.

Preferred as additives are polyethylene, fatty acid alkanolamides and polyurethanes, especially polyethylene and fatty acid alkanolamides. Highly preferred are polyethylene.

A highly preferred fabric softener composition used according to the present invention comprises:

- a) 0.01 to 70 % by weight based on the total weight of the composition of a polyorganosiloxane, or a mixture thereof;
- b) 0.2 to 25 % by weight based on the total weight of an emulsifier, or a mixture thereof;
- c) 0.01 to 25 % by weight, especially 0.01 to 15 % by weight, based on the total weight of at least one additive selected from the group consisting of a polyethylene, a fatty acid alkanolamide, a polysilicic acid, or a polyurethane, and
- d) water to 100 %.

The fabric softener compositions can be prepared as follows:

Firstly, emulsions of the polyorganosiloxane are prepared. The polyorganosiloxane and polyethylene, fatty acid alkanol amide, polysilicic acid or polyurethane are emulsified in water using one or more surfactants and shear forces, e.g. by means of a colloid mill. Suitable surfactants are described above. The components may be emulsified individually before being mixed together, or emulsified together after the components have been mixed. The surfactant(s) is/are used in customary amounts known to the person skilled in the art and can be added either to the polyorganosiloxane or to the water prior to emulsification. Where appropriate, the emulsification operation can be carried out at elevated temperature. The fabric softener composition according to the invention is usually, but not exclusively, prepared by firstly stirring the active substance, i.e. the hydrocarbon based fabric softening component, in the molten state into water, then, where required, adding further desired additives and, finally, after cooling, adding the polyorganosiloxane emulsion.

The fabric softener composition can, for example, be prepared by mixing a preformulated fabric softener with an emulsion comprising the polyorganosiloxane and the additive.

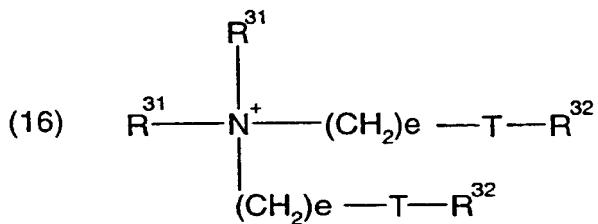
The fabric softening components can be conventional hydrocarbon based fabric softening components known in the art.

Hydrocarbon fabric softeners suitable for use herein are selected from the following classes of compounds:

(i) Cationic quaternary ammonium salts. The counter ion of such cationic quaternary ammonium salts may be a halide, such as chloride or bromide, methyl sulphate, or other ions well known in the literature. Preferably the counter ion is methyl sulfate or any alkyl sulfate or any halide, methyl sulfate being most preferred for the dryer-added articles of the invention.

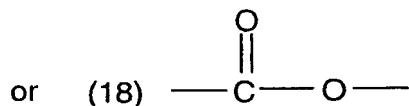
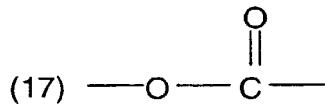
Examples of cationic quaternary ammonium salts include but are not limited to:

(1) Acyclic quaternary ammonium salts having at least two C<sub>8</sub> to C<sub>30</sub>, preferably C<sub>12</sub> to C<sub>22</sub> alkyl or alkenyl chains, such as: ditallowdimethyl ammonium methylsulfate, di(hydrogenated tallow)dimethyl ammonium methylsulfate, distearyldimethyl ammonium methylsulfate, dicocodimethyl ammonium methylsulfate and the like. It is especially preferred if the fabric softening compound is a water insoluble quaternary ammonium material which comprises a compound having two C<sub>12</sub> to C<sub>18</sub> alkyl or alkenyl groups connected to the molecule via at least one ester link. It is more preferred if the quaternary ammonium material has two ester links present. An especially preferred ester-linked quaternary ammonium material for use in the invention can be represented by the formula:



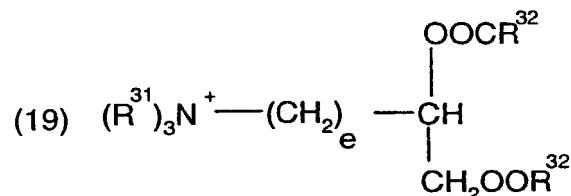
wherein each R<sup>31</sup> group is independently selected from C<sub>1</sub> to C<sub>4</sub> alkyl, hydroxyalkyl or C<sub>2</sub> to C<sub>4</sub> alkenyl groups; T is either

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and wherein each  $R^{32}$  group is independently selected from  $C_8$  to  $C_{28}$  alkyl or alkenyl groups; and  $e$  is an integer from 0 to 5.

A second preferred type of quaternary ammonium material can be represented by the formula:



wherein  $R^{31}$ ,  $e$  and  $R^{32}$  are as defined above.

(2) Cyclic quaternary ammonium salts of the imidazolinium type such as di(hydrogenated tallow)dimethyl imidazolinium methylsulfate, 1-ethylene-bis(2-tallow-1-methyl) imidazolinium methylsulfate and the like;

(3) Diamido quaternary ammonium salts such as: methyl-bis(hydrogenated tallow amidoethyl)-2-hydroxethyl ammonium methyl sulfate, methyl bi(tallowamidoethyl)-2-hydroxypropyl ammonium methylsulfate and the like;

(4) Biodegradable quaternary ammonium salts such as N,N-di(tallowoyl-oxy-ethyl)-N,N-dimethyl ammonium methyl sulfate and N,N-di(tallowoyl-oxy-propyl)-N,N-dimethyl ammonium methyl sulfate. Biodegradable quaternary ammonium salts are described, for example, in U.S. Patents 4,137,180, 4,767,547 and 4,789,491 incorporated by reference herein.

Preferred biodegradable quaternary ammonium salts include the biodegradable cationic diester compounds as described in U.S. Patent 4,137,180, herein incorporated by reference.

(ii) Tertiary fatty amines having at least one and preferably two C8 to C30, preferably C12 to C22 alkyl chains. Examples include hardened tallow-di-methylamine and cyclic amines such as 1-(hydrogenated tallow)amidoethyl-2-(hydrogenated tallow) imidazoline. Cyclic amines which may be employed for the compositions herein are described in U.S. Patent 4,806,255 incorporated by reference herein.

(iii) Carboxylic acids having 8 to 30 carbons atoms and one carboxylic group per molecule. The alkyl portion has 8 to 30, preferably 12 to 22 carbon atoms. The alkyl portion may be linear or branched, saturated or unsaturated, with linear saturated alkyl preferred. Stearic acid is a preferred fatty acid for use in the composition herein. Examples of these carboxylic acids are commercial grades of stearic acid and palmitic acid, and mixtures thereof which may contain small amounts of other acids.

(iv) Esters of polyhydric alcohols such as sorbitan esters or glycerol stearate. Sorbitan esters are the condensation products of sorbitol or iso-sorbitol with fatty acids such as stearic acid. Preferred sorbitan esters are monoalkyl. A common example of sorbitan ester is SPAN 60 (ICI) which is a mixture of sorbitan and isosorbide stearates.

(v) Fatty alcohols, ethoxylated fatty alcohols, alkyphenols, ethoxylated alkyphenols, ethoxylated fatty amines, ethoxylated monoglycerides and ethoxylated diglycerides.

(vi) Mineral oils, and polyols such as polyethylene glycol.

These softeners are more definitively described in U.S. Patent 4,134,838 the disclosure of which is incorporated by reference herein. Preferred fabric softeners for use herein are acyclic quaternary ammonium salts. Di(hydrogenated)tallowdimethyl ammonium methylsulfate is most widely used for dryer articles of this invention. Mixtures of the above mentioned fabric softeners may also be used.

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The fabric softening composition employed in the present invention usually contains about 0.1% to about 95% of the fabric softening component. Preferably from about 2% to about 70% and most preferably from about 2% to about 30% of the fabric softening component is employed herein to obtain optimum softening at minimum cost. When the fabric softening component includes a quaternary ammonium salt, the salt is used in the amount of about 2% to about 70%, preferably about 2% to about 30%.

The fabric softener composition may also comprise additives which are customary for standard commercial liquid rinse conditioners, for example alcohols, such as ethanol, n-propanol, i-propanol, polyhydric alcohols, for example glycerol and propylene glycol; amphoteric and nonionic surfactants, for example carboxyl derivatives of imidazole, oxyethylated fatty alcohols, hydrogenated and ethoxylated castor oil, alkyl polyglycosides, for example decyl polyglucose and dodecylpolyglucose, fatty alcohols, fatty acid esters, fatty acids, ethoxylated fatty acid glycerides or fatty acid partial glycerides; also inorganic or organic salts, for example water-soluble potassium, sodium or magnesium salts, non-aqueous solvents, pH buffers, perfumes, dyes, hydrotropic agents, antifoams, anti redeposition agents, polymeric or other thickeners, enzymes, optical brighteners, antishrink agents, stain removers, germicides, fungicides, antioxidants and corrosion inhibitors.

These fabric softener compositions are traditionally prepared as dispersions containing for example up to 20 % by weight of active material in water. They have a turbid appearance. However, alternative formulations usually containing actives at levels of 5 to 40 % along with solvents can be prepared as microemulsions which have a clear appearance (as to the solvents and the formulations see for example US-A-5,543,067 und WO-A-98/17757). The additives and polyorganosiloxanes of the present invention can be used for such compositions although it will be necessary to use them in microemulsion form to preserve the clear appearance of the fabric softener compositions which are microemulsions.

Another aspect of the invention is a tumble dryer sheet article. The conditioning composition of the present invention may be coated onto a flexible substrate which carries a fabric conditioning amount of the composition and is capable of releasing the composition at dryer operating temperatures. The conditioning composition in turn has a preferred melting (or softening) point of about 25°C to about 150°C.

The fabric conditioning composition which may be employed in the invention is coated onto a dispensing means which effectively releases the fabric conditioning composition in a tumble dryer. Such dispensing means can be designed for single usage or for multiple uses. One such multi-use article comprises a sponge material releasably enclosing enough of the conditioning composition to effectively impart fabric softness during several drying cycles. This multi-use article can be made by filling a porous sponge with the composition. In use, the composition melts and leaches out through the pores of the sponge to soften and condition fabrics. Such a filled sponge can be used to treat several loads of fabrics in conventional dryers, and has the advantage that it can remain in the dryer after use and is not likely to be misplaced or lost.

Another article comprises a cloth or paper bag releasably enclosing the composition and sealed with a hardened plug of the mixture. The action and heat of the dryer opens the bag and releases the composition to perform its softening.

A highly preferred article comprises the inventive compositions releasably affixed to a flexible substrate such as a sheet of paper or woven or non-woven cloth substrate. When such an article is placed in an automatic laundry dryer, the heat, moisture, distribution forces and tumbling action of the dryer removes the composition from the substrate and deposits it on the fabrics.

The sheet conformation has several advantages. For example, effective amounts of the compositions for use in conventional dryers can be easily absorbed onto and into the sheet substrate by a simple dipping or padding process. Thus, the end user need not measure the amount of the composition necessary to obtain fabric softness and other benefits. Additionally, the flat configuration of the sheet provides a large surface area which results in efficient release and distribution of the materials onto fabrics by the tumbling action of the dryer.

The substrates used in the articles can have a dense, or more preferably, open or porous structure. Examples of suitable materials which can be used as substrates herein include paper, woven cloth, and non-woven cloth. The term "cloth" herein means a woven or non-woven substrate for the articles of manufacture, as distinguished from the term "fabric" which encompasses the clothing fabrics being dried in an automatic dryer.

It is known that most substances are able to absorb a liquid substance to some degree; however, the term "absorbent", as used herein, is intended to mean a substrate with an absorbent capacity (i.e., a parameter representing a substrates ability to take up and retain a liquid) from 4 to 12, preferably 5 to 7 times its weight of water.

If the substrate is a foamed plastics material, the absorbent capacity is preferably in the range of 15 to 22, but some special foams can have an absorbent capacity in the range from 4 to 12.

Determination of absorbent capacity values is made by using the capacity testing procedures described in U.S. Federal Specifications (UU-T-595b), modified as follows:

1. tap water is used instead of distilled water;
2. the specimen is immersed for 30 seconds instead of 3 minutes;
3. draining time is 15 seconds instead of 1 minute; and
4. the specimen is immediately weighed on a torsion balance having a pan with turned-up edges.

Absorbent capacity values are then calculated in accordance with the formula given in said Specification. Based on this test, one-ply, dense bleached paper (e.g., Kraft or bond having a basis weight of about 32 pounds per 3,000 square feet) has an absorbent capacity of 3.5 to 4; commercially available household one-ply towel paper has a value of 5 to 6; and commercially available two-ply household towelling paper has a value of 7 to about 9.5.

Suitable materials which can be used as a substrate in the invention herein include, among others, sponges, paper, and woven and non-woven cloth, all having the necessary absorbency requirements defined above.

The preferred non-woven cloth substrates can generally be defined as adhesively bonded fibrous or filamentous products having a web or carded fiber structure (where the fiber strength is suitable to allow carding), or comprising fibrous mats in which the fibers or filaments are distributed haphazardly or in random array (i.e. an array of fibers is a carded web wherein partial orientation of the fibers is frequently present, as well as a completely haphazard distributional orientation), or substantially aligned. The fibers or filaments can be

natural (e.g. wool, silk, jute, hemp, cotton, linen, sisal, or ramie) or synthetic (e.g. rayon, cellulose ester, polyvinyl derivatives, polyolefins, polyamides, or polyesters).

The preferred absorbent properties are particularly easy to obtain with non-woven cloths and are provided merely by building up the thickness of the cloth, i.e., by superimposing a plurality of carded webs or mats to a thickness adequate to obtain the necessary absorbent properties, or by allowing a sufficient thickness of the fibers to deposit on the screen. Any diameter or denier of the fiber (generally up to about 10 denier) can be used, inasmuch as it is the free space between each fiber that makes the thickness of the cloth directly related to the absorbent capacity of the cloth, and which, further, makes the non-woven cloth especially suitable for impregnation with a composition by means of intersectional or capillary action. Thus, any thickness necessary to obtain the required absorbent capacity can be used.

When the substrate for the composition is a non-woven cloth made from fibers deposited haphazardly or in random array on the screen, the articles exhibit excellent strength in all directions and are not prone to tear or separate when used in the automatic clothes dryer.

Preferably, the non-woven cloth is water-laid or air-laid and is made from cellulosic fibers, particularly from regenerated cellulose or rayon. Such non-woven cloth can be lubricated with any standard textile lubricant.

Preferably, the fibers are from 5mm to 50mm in length and are from 1.5 to 5 denier. Preferably, the fibers are at least partially orientated haphazardly, and are adhesively bonded together with a hydrophobic or substantially hydrophobic binder-resin. Preferably, the cloth comprises about 70% fiber and 30% binder resin polymer by weight and has a basis weight of from about 18 to 45g per square meter.

In applying the fabric conditioning composition to the absorbent substrate, the amount impregnated into and/or coated onto the absorbent substrate is conveniently in the weight ratio range of from about 10:1 to 0.5:1 based on the ratio of total conditioning composition to dry, untreated substrate (fiber plus binder). Preferably, the amount of the conditioning composition ranges from about 5:1 to about 1:1, most preferably from about 3:1 to 1:1, by weight of the dry untreated substrate.

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According to one preferred embodiment of the invention, the dryer sheet substrate is coated by being passed over a rotogravure applicator roll. In its passage over this roll, the sheet is coated with a thin, uniform layer of molten fabric softening composition contained in a rectangular pan at a level of about 15g per square yard. Passage for the substrate over a cooling roll then solidifies the molten softening composition to a solid. This type of applicator is used to obtain a uniform homogeneous coating across the sheet.

Following application of the liquefied composition, the articles are held at room temperature until the composition substantially solidifies. The resulting dry articles, prepared at the composition substrate ratios set forth above, remain flexible; the sheet articles are suitable for packaging in rolls. The sheet articles can optionally be slitted or punched to provide a non-blocking aspect at any convenient time if desired during the manufacturing process.

The fabric conditioning composition employed in the present invention includes certain fabric softeners which can be used singly or in admixture with each other.

Examples of suitable textile fibre materials which can be treated with the fabric softener composition are materials made of silk, wool, polyamide, acrylics or polyurethanes, and, in particular, cellulosic fibre materials of all types. Such fibre materials are, for example, natural cellulose fibres, such as cotton, linen, jute and hemp, and regenerated cellulose. Preference is given to textile fibre materials made of cotton. The fabric softener compositions are also suitable for hydroxyl-containing fibres which are present in mixed fabrics, for example mixtures of cotton with polyester fibres or polyamide fibres.

A better understanding of the present invention and of its many advantages will be had by referring to the following Examples, given by way of illustration. The percentages given in the examples are percentages by weight.

Example 1 (preparation of the rinse conditioners)

The liquid rinse conditioners are prepared by using the procedure described below. This type of fabric rinse conditioners is normally known under the name of "triple strength" or "triple fold" formula.

75 % by weight of the total amount of water is heated to 40°C. The molten fabric softener

di-(palmcarboxyethyl)-hydroxyethyl-methylammonium-methosulfate (or Rewoquat WE 38 DPG available from Witco) is added to the heated water under stirring and the mixture is stirred for 1 hour at 40°C. Afterwards the aqueous softener solution is cooled down to below 30°C while stirring. When the solution cools down sufficiently magnesium chloride is added and the pH is adjusted to 3.2 with 0.1 N hydrochloric acid. The formulation is then filled up with water to 100%.

The rinse conditioner formulation as described above was used as a base formulation. In a final step the fabric softener is mixed with a separately prepared polyorganosiloxane /additive emulsion. The fabric softener formulations used in the following examples are listed in the following Table 1.

**Table 1** (rinse conditioner formulations used in the application test for 1 kg wash load)

Rinse conditioner formulation	Polyorgano-siloxane emulsion (calculated on solid content of the emulsion)	Fabric softener Base Formulation	pH
0 (Reference)	-----	13.3 g	3.2
A	0.2 g of Type I	13.3 g	3.2
B	0.2 g of Type II	13.3 g	3.2
C	0.2 g of Type III	13.3 g	3.2
D	0.2 g of Type IV	13.3 g	3.2
E	0.2 g of Type V	13.3 g	3.2
F	0.2 g of Type VI	13.3 g	3.2
G	0.2 g of Type VII	13.3 g	3.2
H	0.2 g of Type VIII	13.3 g	3.2
I	0.2 g of Type IX	13.3 g	3.2
J	0.2 g of Type X	13.3 g	3.2
K	0.2 g of Type XV	13.3 g	3.2
L	0.2 g of Type XVI	13.3 g	3.2

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Types of polyorganosiloxane emulsions used

Type I

- Polyorganosiloxane of general formula (1), wherein R<sub>1</sub> is -OH, R<sub>3</sub> is -CH<sub>3</sub>, X + Y = 300-1500, % nitrogen (with respect to silicone) = 0
- 4.1% of an emulsifier
- 7.8% of a fatty acid dialkanolamide of formula (15a), wherein R<sub>34</sub>, R<sub>38</sub>, R<sub>38'</sub> and R<sub>38''</sub> are hydrogen or -CH<sub>2</sub>OH
- solid content of the emulsion measured by evaporation at 120°C = 23.5-25.5%
- water content = 75%

Type II

- Polyorganosiloxane of general formula (1), wherein R<sub>1</sub> is -CH<sub>3</sub>, R<sub>3</sub> is -CH<sub>2</sub>CH<sub>2</sub>CH<sub>2</sub>NH<sub>2</sub>, X + Y = 150-300, % nitrogen (with respect to silicone) = 0.07
- 11% of an emulsifier
- 0.65% of an emulsifiable oxidised polyethylene which has a density of 0.95 to 1.05 g/cm<sup>3</sup> at 20°C, a drop point of 100-150°C, an acid number of 10 to 60 and a saponification number of 15 to 80
- solid content of the emulsion measured by evaporation at 120°C = 27.0-30.0%
- water content = 60.7%

Type III

- Polyorganosiloxane of general formula (1), wherein R<sub>1</sub> is -CH<sub>3</sub>, R<sub>3</sub> is -CH<sub>2</sub>CH<sub>2</sub>CH<sub>2</sub>NH<sub>2</sub>, X + Y = 150-300, % nitrogen (with respect to silicone) = 0.02
- 2.9% of an emulsifier
- 0.23% of a fatty acid dialkanolamide of formula (15a), wherein R<sub>34</sub>, R<sub>38</sub>, R<sub>38'</sub> and R<sub>38''</sub> are hydrogen or -CH<sub>2</sub>OH
- solid content of the emulsion measured by evaporation at 120°C = 7.0-8.0%
- water content = 89.4%

Type IV

- Polyorganosiloxane of general formula (1), wherein R<sub>1</sub> is -OH,

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$R_3$  is  $-\text{CH}_2\text{CH}_2\text{CH}_2\text{N}(\text{H})(\text{CH}_2\text{CH}_2\text{NH}_2)$ ,  $X + Y = 300-1500$ ,

% nitrogen (with respect to silicone) = 0.03

- 3.6% of an emulsifier

- 14% of an emulsifiable oxidised polyethylene which has a density of 0.95 to 1.05 g/cm<sup>3</sup> at 20°C, a drop point of 100-150°C, an acid number of 10 to 60 and a saponification number of 15 to 80

- solid content of the emulsion measured by evaporation at 120°C = 23.0-25.0%

- water content = 73.7%

#### Type V

- Polyorganosiloxane of general formula (1), wherein  $R_1$  is -OH,

$R_3$  is  $-\text{CH}_2\text{CH}_2\text{CH}_2\text{N}(\text{H})(\text{CH}_2\text{CH}_2\text{NH}_2)$ ,  $X + Y = 300-1500$ ,

% nitrogen (with respect to silicone) = 0.11

- 4.3% of an emulsifier

- 0.3% of a fatty acid monoalkanolamide of formula (15b), wherein  $R_{34}$  is hydrogen and  $R_{37}$  is hydrogen or a radical of formula  $-\text{C}(\text{O})\text{R}_{36}$

- solid content of the emulsion measured by evaporation at 120°C = 37.0-39.0%

- water content = 60.7%

#### Type VI

- Polyorganosiloxane of general formula (1), wherein  $R_1$  is  $-\text{CH}_3$ ,

$R_3$  is  $-\text{CH}_2\text{CH}_2\text{CH}_2\text{N}(\text{H})(\text{CH}_2\text{CH}_2\text{NH}_2)$ ,  $X + Y = 150-300$ ,

% nitrogen (with respect to silicone) = 0.12

- 11% of an emulsifier

- 0.3% of a fatty acid dialkanolamide of formula (15a), wherein  $R_{34}$ ,  $R_{38}$ ,  $R_{38}'$  and  $R_{38}''$  are hydrogen or  $-\text{CH}_2\text{OH}$

- solid content of the emulsion measured by evaporation at 120°C = 24.0-26.0%

- water content = 72.1%

#### Type VII

- Polyorganosiloxane of general formula (8), wherein  $R_{17}$  is  $-\text{CH}_3$ ,  $R_3$  is  $\text{CH}_3$ ,

$R_{19}$  is a polyethylenoxide radical,  $X^1 + Y^1 + S = 40-150$ ,

% nitrogen (with respect to silicone) = 0

- 2% of an emulsifier

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- 0.15% of an emulsifiable oxidised polyethylene which has a density of 0.95 to 1.05 g/cm<sup>3</sup> at 20°C, a drop point of 100-150°C, an acid number of 10 to 60 and a saponification number of 15 to 80
- solid content of the emulsion measured by evaporation at 120°C = 23.0-25.0%
- water content = 74.9%

#### Type VIII

- Polyorganosiloxane of general formula (8), wherein R<sub>17</sub> is -CH<sub>3</sub>, R<sub>3</sub> is -CH<sub>2</sub>CH<sub>2</sub>CH<sub>2</sub>NH<sub>2</sub>, R<sub>19</sub> is a polyethylene/polypropyleneoxide radical, X<sup>1</sup> + Y<sup>1</sup> + S = 150-300
- % nitrogen (with respect to silicone) = 0.044
- 2.5% of an emulsifier
- 2.94% of an emulsifiable oxidised polyethylene which has a density of 0.95 to 1.05 g/cm<sup>3</sup> at 20°C, a drop point of 100-150°C, an acid number of 10 to 60 and a saponification number of 15 to 80
- solid content of the emulsion measured by evaporation at 120°C = 15.5-17.5%
- water content = 80.4%

#### Type IX

- Polyorganosiloxane of general formula (8), wherein R<sub>17</sub> is -CH<sub>3</sub>, R<sub>3</sub> is -CH<sub>2</sub>CH<sub>2</sub>CH<sub>2</sub>NH<sub>2</sub>, R<sub>19</sub> is a polyethylene/polypropyleneoxide radical, X<sup>1</sup> + Y<sup>1</sup> + S = 150-300
- % nitrogen (with respect to silicone) = 0.07
- 3.5% of an emulsifier
- 1.5% of a fatty acid dialkanolamide of formula (15a), wherein R<sub>34</sub>, R<sub>38</sub>, R<sub>38'</sub> and R<sub>38''</sub> are hydrogen or -CH<sub>2</sub>OH
- solid content of the emulsion measured by evaporation at 120°C = 19.5-21.5%
- water content = 73%

#### Type X

- Polyorganosiloxane of general formula (1), wherein R<sub>1</sub> is -CH<sub>3</sub>, R<sub>3</sub> is C<sub>18</sub>alkoxy, X + Y = 40-150,
- % nitrogen (with respect to silicone) = 0
- 3.2% of an emulsifier

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- 1.5% of an emulsifiable oxidised polyethylene which has a density of 0.95 to 1.05 g/cm<sup>3</sup> at 20°C, a drop point of 100-150°C, an acid number of 10 to 60 and a saponification number of 15 to 80
- solid content of the emulsion measured by evaporation at 120°C = 34.0-35.5%
- water content = 61.4%

#### Type XI

- Polyorganosiloxane of general formula (8), wherein R<sub>17</sub> is -CH<sub>3</sub>, R<sub>3</sub> is -CH<sub>3</sub>, R<sub>19</sub> is a polyethylene/polypropyleneoxide radical,  
 $X^1 + Y^1 + S = 150-300$   
% nitrogen (with respect to silicone) = 0
- 3% of an emulsifier
- 0.15% of an emulsifiable oxidised polyethylene which has a density of 0.95 to 1.05 g/cm<sup>3</sup> at 20°C, a drop point of 100-150°C, an acid number of 10 to 60 and a saponification number of 15 to 80
- solid content of the emulsion measured by evaporation at 120°C = 30-32%
- water content = 63.9%

#### Type XII

- Polyorganosiloxane of general formula (11), j = 300,  
% nitrogen (with respect to silicone) = 0.04-0.06
- 9% of an emulsifier
- solid content of the emulsion measured by evaporation at 120°C = 21-23%
- water content = 73%

#### Type XIII

- Polyorganosiloxane of general formula (1), wherein R<sub>1</sub> is -OH, R<sub>3</sub> is -CH<sub>2</sub>CH<sub>2</sub>CH<sub>2</sub>N(H)(CH<sub>2</sub>CH<sub>2</sub>NH<sub>2</sub>), X + Y = 300-1500,  
% nitrogen (with respect to silicone) = 0.1
- 4.2% of an emulsifier
- 6.2% of a fatty acid monoalkanolamide of formula (15b), wherein R<sub>34</sub> is hydrogen and R<sub>37</sub> is hydrogen or a radical of formula -C(O)R<sub>36</sub>
- solid content of the emulsion measured by evaporation at 120°C = 38-40%
- water content = 60%

Type XIV

- Polyorganosiloxane of general formula (8), wherein R<sub>17</sub> is -CH<sub>3</sub>, R<sub>3</sub> is -CH<sub>2</sub>CH<sub>2</sub>CH<sub>2</sub>NH<sub>2</sub>, R<sub>19</sub> is a polyethylenoxide radical, X<sup>1</sup> + Y<sup>1</sup> + S = 40-150,
- % nitrogen (with respect to silicone) = 0.04
- 7.2% of an emulsifier
- solid content of the emulsion measured by evaporation at 120°C = 54-56%
- water content = 38.1%

Type XV

Mixture of 1 part of emulsion Type XIII and 9 parts of emulsion Type XIV.

Type XVI

Mixture of 1 part of emulsion Type XI and 2 parts of emulsion Type XII.

Example 2 (Antipilling)

The formulated rinse conditioners (see Table 1) are applied according to the following procedure:

Textile swatches are washed in a washing machine, rinsed and dried. The antipilling properties are evaluated after 1 wash/rinse-cycle.

The textile used is: Cotton knit: 163 g/m<sup>2</sup>, bleached

The textile is finished with a resin according to Oekotex Standard 100:

30 g/l of modified dimethyloldihydroxyethylene urea ( 60% active material)

9 g/l Magnesiumchloride (with 6 H<sub>2</sub>O)

padding with a pick-up of approximately 60%

Drying at about 110 - 120 °C in a oven followed by a 4 minute curing step at 145°C

Cotton knit swatches of size of 50 cm by 40 cm are washed together with ballast material (cotton and cotton/polyester) in a AEG Oeko Lavamat 73729 washing machine maintaining the washing temperature at 40°C . The total fabric load of 1 kg is washed for 15 minutes with 33 g of ECE Color Fastness Test Detergent 77 (Formulation January 1977, according to ISO 105-CO6). The rinse conditioner formulation as described in Table 1 is applied in the last

rinse cycle at 20°C. After rinsing with the formulation the textile swatches are dried on a washing line at ambient temperature.

Evaluation of the pilling

The pilling of the treated swatches is tested and evaluated according to a method described under point 3 (SN 198525, 1990). A number of 1 is assigned to a very strong pilling, a number of 5 reflects no or very slight pilling.

The following results (evaluated after 125, 250 and 500 rotations) have been found :

Table 2 (Results of pilling tests)

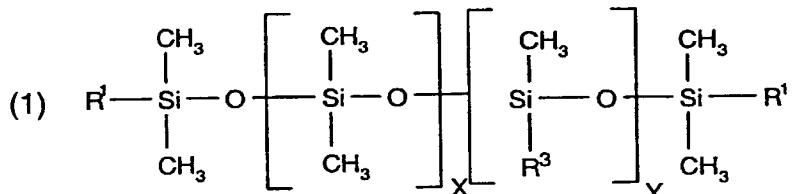
Number of rotations	125	250	500
Reference	3.3	3.0	2.8
A	4.8	4.0	3.5
B	4.3	4.0	3.2
C	4.3	3.3	3.2
D	4.5	4.0	3.5
E	4.7	4.5	3.5
F	4.3	4.0	3.5
G	4.0	3.5	3.2
H	4.5	4.0	3.7
I	4.8	4.2	4.2
J	4.0	3.3	3.2
K	4.8	4.7	3.8
L	4.0	3.5	3.0

These results show a markedly improvement resistance to pilling when textile fabric material is treated with compositions of the present invention.

## WHAT IS CLAIMED IS:

1. A method of use of a softener composition for the antipilling treatment of textile fibre materials in domestic applications, which softener composition comprises:

- A) a fabric softener;
- B) at least one additive selected from the group consisting of
  - a) a polyethylene, or a mixture thereof,
  - b) a fatty acid alkanolamide, or a mixture thereof,
  - c) a polysilicic acid, or a mixture thereof, and
  - d) a polyurethane, or a mixture thereof; and
- C) a dispersed polyorganosiloxane of formula (1)

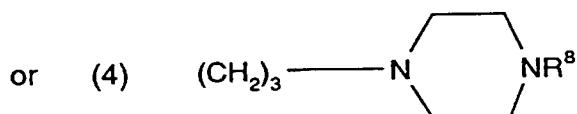
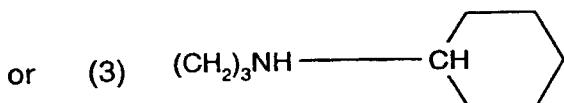
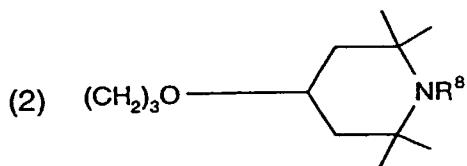


wherein

$\text{R}^1$  is  $\text{OH}$ ,  $\text{OR}^2$  or  $\text{CH}_3$

$\text{R}^2$  is  $\text{CH}_3$  or  $\text{CH}_2\text{CH}_3$

$\text{R}^3$  is  $\text{C}_1\text{-C}_{20}$ alkoxy,  $\text{CH}_3$ ,  $\text{CH}_2\text{CHR}^4\text{CH}_2\text{NHR}^5$ , or  $\text{CH}_2\text{CHR}^4\text{CH}_2\text{N}(\text{COCH}_3)\text{R}^5$



$\text{R}^4$  is  $\text{H}$  or  $\text{CH}_3$

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$R^5$  is H,  $CH_2CH_2NHR^6$ ,  $C(=O)-R^7$  or  $(CH_2)_z-CH_3$

$z$  is 0 to 7

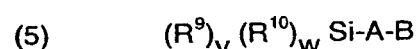
$R^6$  is H or  $C(=O)-R^7$

$R^7$  is  $CH_3$ ,  $CH_2CH_3$  or  $CH_2CH_2CH_2OH$

$R^8$  is H or  $CH_3$

the sum of X and Y is 40 to 4000;

or a dispersed polyorganosiloxane which comprises at least one unit of the formula (5)



wherein

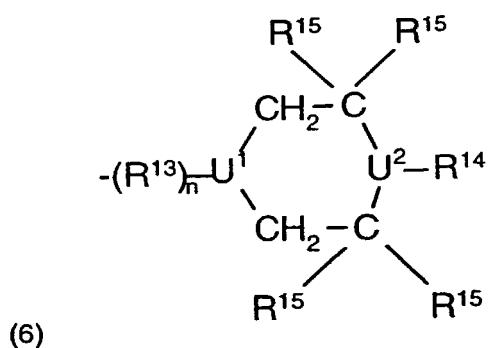
$R^9$  is  $CH_3$ ,  $CH_3CH_2$  or Phenyl

$R^{10}$  is  $-O-Si$  or  $-O-R^9$

the sum of  $v$  and  $w$  equals 3, and  $v$  does not equal 3

$A = -CH_2CH(R^{11})(CH_2)_K$

$B = -NR^{12}((CH_2)_l-NH)_mR^{12}$ , or



$n$  is 0 or 1

when  $n$  is 0,  $U^1$  is N, when  $n$  is 1,  $U^1$  is CH

$l$  is 2 to 8

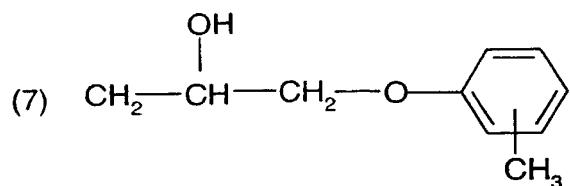
$k$  is 0 to 6

$m$  is 0 to 3

$R^{11}$  is H or  $CH_3$

$R^{12}$  is H,  $C(=O)-R^{16}$ ,  $CH_2(CH_2)_pCH_3$  or

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p is 0 to 6

R<sup>13</sup> is NH, O, OCH<sub>2</sub>CH(OH)CH<sub>2</sub>N(Butyl), OOCN(Butyl)

R<sup>14</sup> is H, linear or branched C<sub>1</sub>-C<sub>4</sub> alkyl, Phenyl or CH<sub>2</sub>CH(OH)CH<sub>3</sub>

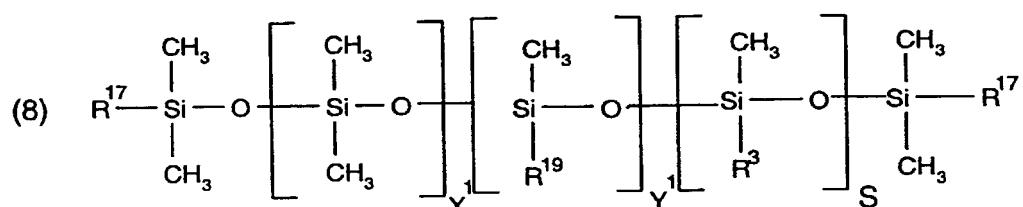
R<sup>15</sup> is H or linear or branched C<sub>1</sub>-C<sub>4</sub> alkyl

R<sup>16</sup> is CH<sub>3</sub>, CH<sub>2</sub>CH<sub>3</sub> or (CH<sub>2</sub>)<sub>q</sub>OH

q is 1 to 6

U<sup>2</sup> is N or CH;

or a dispersed polyorganosiloxane of the formula (8)



wherein

R<sup>3</sup> is as previously defined

R<sup>17</sup> is OH, OR<sup>18</sup> or CH<sub>3</sub>

R<sup>18</sup> is CH<sub>3</sub> or CH<sub>2</sub>CH<sub>3</sub>

R<sup>19</sup> is R<sup>20</sup>-(EO)<sub>m</sub>-(PO)<sub>n</sub>-R<sup>21</sup>

m is 3 to 25

n is 0 to 10

R<sup>20</sup> is the direct bond or CH<sub>2</sub>CH(R<sup>22</sup>)(CH<sub>2</sub>)<sub>p</sub>R<sup>23</sup>

p is 1 to 4

R<sup>21</sup> is H, R<sup>24</sup>, CH<sub>2</sub>CH(R<sup>22</sup>)NH<sub>2</sub> or CH(R<sup>22</sup>)CH<sub>2</sub>NH<sub>2</sub>

R<sup>22</sup> is H or CH<sub>3</sub>

R<sup>23</sup> is O or NH

R<sup>24</sup> is linear or branched C<sub>1</sub>-C<sub>8</sub> alkyl or Si(R<sup>25</sup>)<sub>3</sub>

R<sup>25</sup> is R<sup>24</sup>, OCH<sub>3</sub> or OCH<sub>2</sub>CH<sub>3</sub>

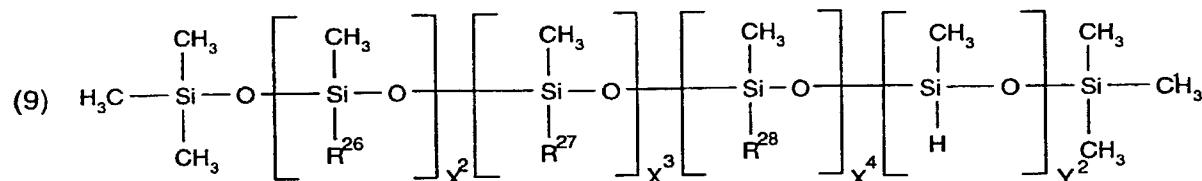
EO is -CH<sub>2</sub>CH<sub>2</sub>O-

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PO is  $-\text{CH}(\text{CH}_3)\text{CH}_2\text{O}-$  or  $-\text{CH}_2\text{CH}(\text{CH}_3)\text{O}-$

the sum of  $X_1, Y_1$  and S is 20 to 1500;

or a dispersed polyorganosiloxane of the formula (9)



wherein

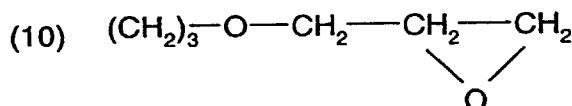
$\text{R}^{26}$  is linear or branched  $\text{C}_1 - \text{C}_{20}$  alkoxy,  $\text{CH}_2\text{CH}(\text{R}^4)\text{R}^{29}$

$\text{R}^4$  is as previously defined

$\text{R}^{29}$  is linear or branched  $\text{C}_1 - \text{C}_{20}$  alkyl

$\text{R}^{27}$  is aryl, aryl substituted by linear or branched  $\text{C}_1 - \text{C}_{10}$  alkyl, linear or branched  $\text{C}_1 - \text{C}_{20}$  alkyl substituted by aryl or aryl substituted by linear or branched  $\text{C}_1 - \text{C}_{10}$  alkyl

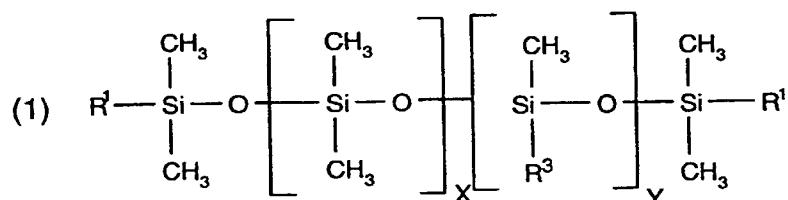
$\text{R}^{28}$  is



the sum of  $X^2, X^3, X^4$  and  $Y^2$  is 20 to 1500, wherein  $X^3, X^4$  and  $Y^2$  may be independently of each other 0;

or a mixture thereof.

2. A method of use according to claim 1 wherein the polyorganosiloxane is of formula (1):



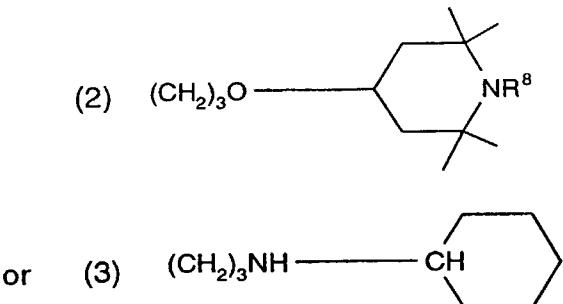
wherein

$\text{R}^1$  is  $\text{OH}, \text{OR}^2$  or  $\text{CH}_3$

$\text{R}^2$  is  $\text{CH}_3$  or  $\text{CH}_2\text{CH}_3$

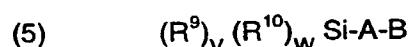
$\text{R}^3$  is  $\text{C}_1\text{-C}_{20}$  alkoxy,  $\text{CH}_3$ ,  $\text{CH}_2\text{CHR}^4\text{CH}_2\text{NHR}^5$ , or

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 $\text{R}^4$  is H or  $\text{CH}_3$  $\text{R}^5$  is H,  $\text{CH}_2\text{CH}_2\text{NHR}^6$ ,  $\text{C}(=\text{O})\text{-R}^7$  $\text{R}^6$  is H or  $\text{C}(=\text{O})\text{-R}^7$  $\text{R}^7$  is  $\text{CH}_3$ ,  $\text{CH}_2\text{CH}_3$  or  $\text{CH}_2\text{CH}_2\text{CH}_2\text{OH}$  $\text{R}^8$  is H or  $\text{CH}_3$ 

the sum of X and Y is 40 to 1500

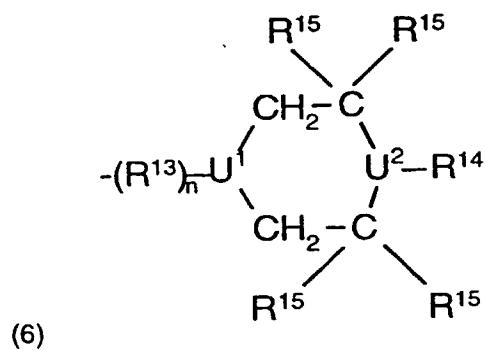
or a dispersed polyorganosiloxane which comprises at least one unit of the formula (5);



wherein

 $\text{R}^9$  is  $\text{CH}_3$ ,  $\text{CH}_3\text{CH}_2$  $\text{R}^{10}$  is  $-\text{O-Si}$  or  $-\text{O-R}^9$ 

the sum of v and w equals 3, and v does not equal 3

 $\text{A} = -\text{CH}_2\text{CH}(\text{R}^{11})(\text{CH}_2)_K$  $\text{B} =$ 

n is 1

U<sup>1</sup> is CH

k is 0 to 6

R<sup>11</sup> is H or CH<sub>3</sub>

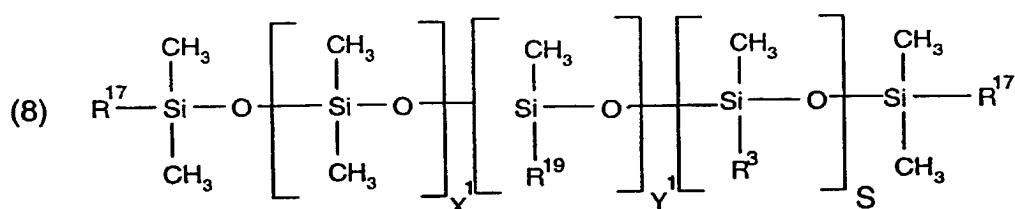
R<sup>13</sup> is OOCN(Butyl)

R<sup>14</sup> is H, linear C<sub>1</sub>-C<sub>4</sub> alkyl, Phenyl

R<sup>15</sup> is H or linear C<sub>1</sub>-C<sub>4</sub> alkyl

U<sup>2</sup> is N

or a dispersed polyorganosiloxane of the formula (8);



wherein

R<sup>3</sup> is as previously defined

R<sup>17</sup> is OH, OR<sup>18</sup> or CH<sub>3</sub>

R<sup>18</sup> is CH<sub>3</sub> or CH<sub>2</sub>CH<sub>3</sub>

R<sup>19</sup> is R<sup>20</sup>-(EO)<sub>m</sub>-(PO)<sub>n</sub>-R<sup>21</sup>

m is 3 to 25

n is 0 to 10

R<sup>20</sup> is the direct bond or CH<sub>2</sub>CH(R<sup>22</sup>)(CH<sub>2</sub>)<sub>p</sub>R<sup>23</sup>

p is 1 to 4

R<sup>21</sup> is H, R<sup>24</sup>, CH<sub>2</sub>CH(R<sup>22</sup>)NH<sub>2</sub> or CH(R<sup>22</sup>)CH<sub>2</sub>NH<sub>2</sub>

R<sup>22</sup> is H or CH<sub>3</sub>

R<sup>23</sup> is O or NH

R<sup>24</sup> is linear or branched C<sub>1</sub>-C<sub>3</sub> alkyl or Si(R<sup>25</sup>)<sub>3</sub>

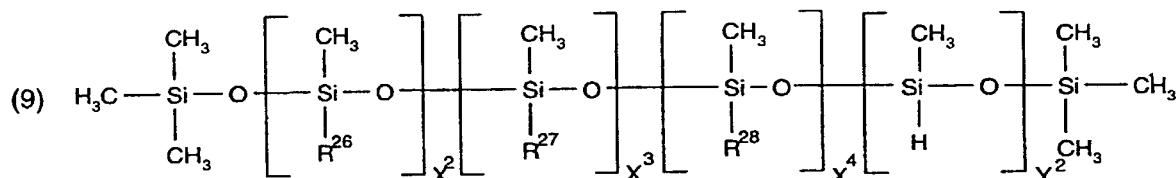
R<sup>25</sup> is R<sup>24</sup>, OCH<sub>3</sub> or OCH<sub>2</sub>CH<sub>3</sub>

EO is -CH<sub>2</sub>CH<sub>2</sub>O-

PO is -CH(CH<sub>3</sub>)CH<sub>2</sub>O- or -CH<sub>2</sub>CH(CH<sub>3</sub>)O-

the sum of X<sub>1</sub>, Y<sub>1</sub> and S is 40 to 1500

or a dispersed polyorganosiloxane of the formula (9);



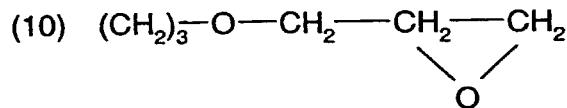
$\text{R}^{26}$  is linear  $\text{C}_1 - \text{C}_{20}$  alkoxy,

$\text{R}^4$  is as previously defined

$\text{R}^{29}$  is linear  $\text{C}_1 - \text{C}_{20}$  alkyl

$\text{R}^{27}$  is,  $\text{CH}_2\text{CH}(\text{R}^4)\text{Phenyl}$

$\text{R}^{28}$  is



the sum of  $\text{X}^2$ ,  $\text{X}^3$ ,  $\text{X}^4$  and  $\text{Y}^2$  is 40 to 1500, wherein  $\text{X}^3$ ,  $\text{X}^4$  and  $\text{Y}^2$  may be independently of each other 0;

or a mixture thereof.

3. A method of use according to claim 1 or 2 wherein a polyorganosiloxane of formula (1) is used, wherein

$\text{R}^1$  is OH or  $\text{CH}_3$ ,

$\text{R}^3$  is  $\text{CH}_3$ ,  $\text{C}_{10}-\text{C}_{20}$  alkoxy or  $\text{CH}_2\text{CHR}^4\text{CH}_2\text{NHR}^5$ ,

$\text{R}^4$  is H,

$\text{R}^5$  is H or  $\text{CH}_2\text{CH}_2\text{NHR}^6$ ,

$\text{R}^6$  is H or  $\text{C}(\text{=O})-\text{R}^7$ , and

$\text{R}^7$  is  $\text{CH}_3$ ,  $\text{CH}_2\text{CH}_3$  or especially  $\text{CH}_2\text{CH}_2\text{CH}_2\text{OH}$ .

4. A method of use according to claim 1 or 2 wherein a polyorganosiloxane of formula (8) is used, wherein

$\text{R}^3$  is  $\text{CH}_3$ ,  $\text{C}_{10}-\text{C}_{20}$  alkoxy or  $\text{CH}_2\text{CHR}^4\text{CH}_2\text{NHR}^5$ ,

$\text{R}^4$  is H,

$\text{R}^5$  is H or  $\text{CH}_2\text{CH}_2\text{NHR}^6$ ,

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R<sup>6</sup> is H or C(=O)-R<sup>7</sup>,

R<sup>7</sup> is CH<sub>2</sub>CH<sub>3</sub>, CH<sub>2</sub>CH<sub>2</sub>CH<sub>2</sub>OH or especially CH<sub>3</sub>, and

R<sub>17</sub> is CH<sub>3</sub> or OH.

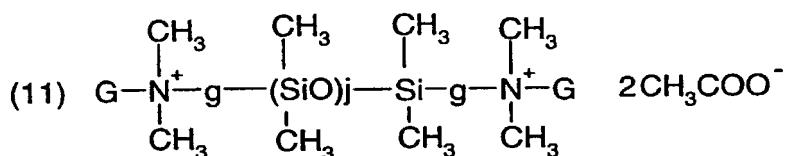
5. A method of use according to claim 1 or 2 wherein a polyorganosiloxane of formula (9) is used, wherein

R<sup>26</sup> is CH<sub>2</sub>CH(R<sup>4</sup>)R<sup>29</sup>,

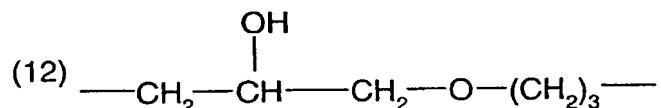
R<sup>4</sup> is H, and

R<sup>27</sup> is 2-phenyl propyl.

6. A method of use according to any of claims 1 to 5 wherein the polyorganosiloxane compositions comprises an additional polyorganosiloxane of the formula (11):



wherein g is



and G is C<sub>1</sub> to C<sub>20</sub> alkyl.

7. A method of use according to any of claims 1 to 6 wherein the composition is a liquid aqueous composition.

8. A method of use according to any of claims 1 to 6 wherein the composition is used in a tumble dryer sheet composition.

9. A method of use according to any of claims 1 to 8 in which the polyorganosiloxane is nonionic or cationic.

10. A method of use according to any of claims 1 to 9 in which the composition has a solids content of 5 to 70 % at a temperature of 120°C.
11. A method of use according to any of claims 1 to 10 in which the composition contains a water content of 25 to 90 % by weight based on the total weight of the composition.
12. A method of use according to any of claims 1 to 11 in which the composition has a pH value from 2 to 7.
13. A method of use according to any of claims 1 to 12 in which the nitrogen content of the aqueous emulsion due to the polyorganosiloxane is from 0 to 0.25 % with respect to the silicon content.
14. A method of use according to any of claims 1 to 13 wherein the composition comprises a polyethylene, a fatty acid alkanolamide or a polyurethane.
15. A method of use according to any of claims 1 to 14 wherein the composition comprises a polyethylene or a fatty acid alkanolamide.
16. A method of use according to any of claims 1 to 15 wherein the composition comprises a fatty acid alkanolamide.
17. A method of use according to any of claims 1 to 15 wherein the composition comprises a polyethylene.
18. A method of use according to any of claims 1 to 17 wherein the composition is prepared by mixing a preformulated fabric softener with an emulsion comprising the polyorganosiloxane and the additive.
19. A method of use according to any of claims 1 to 18 wherein composition has a clear appearance.
20. A method of use according to any of claims 1 to 19 in which the composition comprises:

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- a) 0.01 to 70 % by weight, based on the total weight of the composition, of a polyorganosiloxane, or a mixture thereof;
- b) 0.2 to 25 % by weight based on the total weight of an emulsifier, or a mixture thereof;
- c) 0.01 to 15 % by weight based on the total weight of at least one additive selected from the group consisting of a polyethylene, a fatty acid alkanolamide, a polysilicic acid and a polyurethane, and
- d) water to 100 %.

21. A tumble dryer sheet comprising a composition as defined in claim 1.

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86462 Langweid (DE). **GORETZKI, Ralf** [DE/DE];  
Berliner Allee 22B, 86153 Augsburg (DE). **WEBER, Barbara** [DE/DE]; Schlossweg 1, 79639 Grenzach-Wyhlen (DE). **MARTIN, Emmanuel** [FR/FR]; 4, Cité Beaulieu, F-68300 Saint Louis Neuweg (FR).

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(74) Common Representative: **CIBA SPECIALTY CHEMICALS HOLDING INC.**; Patentabteilung, Klybeckstrasse 141, CH-4057 Basle (CH).

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(71) Applicants (for all designated States except US):  
**CIBA SPECIALTY CHEMICALS HOLDING INC.** [CH/CH]; Klybeckstrasse 141, CH-4057 Basle (CH).  
**CIBA SPEZIALITÄTENCHEMIE PFERSEE GMBH** [DE/DE]; Rehlinger Strasse 1, 86462 Langweid a. Lech (DE).

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(72) Inventors; and  
(75) Inventors/Applicants (for US only): **KVITA, Petr** [CH/CH]; Binningerstrasse 4D, 4153 Reinach (CH). **OTTO, Peter** [US/DE]; Ritterstrasse 26, 79618 Rheinfelden (DE). **DUBINI, Mario** [CH/CH]; Härgelenstrasse 29, CH-4435 Niederdorf (CH). **CHROBACZEK, Harald** [DE/DE]; Oblatterwallstrasse 38, 86153 Augsburg (DE). **GEUBTNER, Michael** [DE/DE]; Aggensteinstrasse 5,

(54) Title: FABRIC SOFTENER COMPOSITIONS

(57) Abstract: The present invention relates to a method of use of a softener composition for the antipilling treatment of textile fibre materials in domestic applications, which softener composition comprises : A) a fabric softener; B) at least one additive selected from the group consisting of a) a polyethylene, or a mixture thereof, b) a fatty acid alkanolamide, or a mixture thereof, c) a polysilicic acid, or a mixture thereof, and d) a polyurethane, or a mixture thereof; and C) a selected polyorganosiloxane compound.

**WO 01/25381 A1**

**DECLARATION AND POWER OF ATTORNEY FOR U.S. PATENT APPLICATIONS**

Original       Supplemental       Substitute       PCT

As a below named inventor, I hereby declare that:

My residence, post office address and citizenship are as stated below next to my name.

I believe I am the original, first and sole inventor (if only one name is listed below) or an original, first and joint inventor (if more than one name is listed below) of the subject matter which is claimed and for which a patent is sought on the invention entitled

**FABRIC SOFTENER COMPOSITIONS**

which is described and claimed in:

the attached specification.

the specification in U.S. Application No. \_\_\_\_\_, filed \_\_\_\_\_, and as amended on \_\_\_\_\_ (if applicable).  
(day/month/year) \_\_\_\_\_ (day/month/year)

the specification in International Application No. \_\_\_\_\_, filed 26/09/00 (if applicable).  
assigned U.S. Application No. \_\_\_\_\_, and as amended  
 under PCT Article 19 on \_\_\_\_\_ (if applicable)  
 under PCT Article 34 on \_\_\_\_\_ (if applicable)  
 and further amended on \_\_\_\_\_ (if applicable)  
(day/month/year)

I hereby state that I have reviewed and understand the contents of the above identified specification, including the claims, as amended by any amendment referred to above.

I acknowledge the duty to disclose all information which is known by me to be material to the patentability of this application as defined in 37 C.F.R. § 1.56.

I hereby claim foreign priority benefits under 35 U.S.C. § 119 (a)-(d) of any foreign application(s) for patent or inventor's certificate or of any PCT international application(s) designating at least one country other than the United States of America listed below and have also identified below any foreign application(s) for patent or inventor's certificate or any PCT international application(s) designating at least one country other than the United States of America relating to this subject matter having a filing date before that of the application on which priority is claimed:

COUNTRY/REGION (OR PCT)	APPLICATION No.	FILING DATE (day/month/year)	PRIORITY CLAIMED	
Europe (designating DE)	99810901.1	05/10/99	<input checked="" type="checkbox"/> Yes	<input type="checkbox"/> No
			<input type="checkbox"/> Yes	<input type="checkbox"/> No
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			<input type="checkbox"/> Yes	<input type="checkbox"/> No
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I hereby claim the benefit under 35 U.S.C. § 119 (e) of any United States provisional application(s) listed below:

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		<input type="checkbox"/> Patented	<input type="checkbox"/> Pending	<input type="checkbox"/> Abandoned
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			<input type="checkbox"/> Pending
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Luther A. R. Hall (Reg. No. 27,337), JoAnn L. Villamizar (Reg. No. 30,598), Kevin T. Mansfield (Reg. No. 31,635), David R. Crichton (Reg. No. 37,300), Michele A. Kovaleski (Reg. No. 37,865) and Tyler A. Stevenson (Reg. No. 46,388).

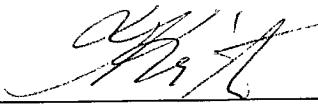
Address all correspondence associated with Customer No. 000324 to **Ciba Specialty Chemicals Corporation, Patent Department, 540 White Plains Road, P.O. Box 2005, Tarrytown, NY 10591-9005.**

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Full name of sole  
or first joint inventor

Petr KVITA

100 Inventor's signature



Date

02/04/02

(day/month/year)

Residence

Binningerstrasse 4D  
4153 Reinach  
Switzerland



Citizenship

Swiss

Post Office Address

same as above

Full name of second joint inventor, if any

Peter OTTO

Inventor's signature



Date

12.3.02

(day/month/year)

200  
Residence

Ritterstrasse 26  
79618 Rheinfelden  
Germany

DEX

Citizenship

American

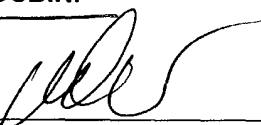
Post Office Address

**same as above**

Full name of third joint inventor, if any

Mario DUBINI

300  
Inventor's signature



Date

12.3.02

(day/month/year)

Residence

Härgelenstrasse 29  
4435 Niederdorf  
Switzerland

CH X

Citizenship

Swiss

Post Office Address

**same as above**

Full name of fourth joint inventor, if any

Harald CHROBACZEK

400  
Inventor's signature



Date

01.03.02

(day/month/year)

Residence

Oblatterwallstrasse 38  
86153 Augsburg  
Germany

DEX

Citizenship

German

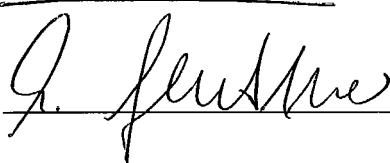
Post Office Address

**same as above**

Full name of fifth  
joint inventor, if any

Michael GEÜBTNER

Inventor's signature



Date 06.03.02  
(day/month/year)

50  
Residence

**Aggensteinstrasse 5  
86462 Langweid DEX**  
Germany

Citizenship

**German**

Post Office Address

**same as above**

---

Full name of sixth  
joint inventor, if any

Ralf GORETZKI

60  
Inventor's signature



Date 06.03.02  
(day/month/year)

60  
Residence

**Kornstrasse 9a  
86391 Stadtbergen DEX**  
Germany

Citizenship

**German**

Post Office Address

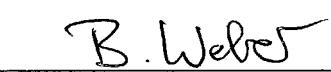
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---

70  
Full name of seventh  
joint inventor, if any

Barbara WEBER

70  
Inventor's signature



Date 13.03.02  
(day/month/year)

Residence

**Schlossweg 1  
79639 Grenzach-Wyhlen DEX**  
Germany

Citizenship

**German**

Post Office Address

**same as above**

Full name of eighth-  
joint inventor, if any

Emmanuel MARTIN

Inventor's signature

E. Martin

Date

13/03/02  
(day/month/year)

Residence

4, Cité Beaulieu  
68300 Saint Louis Neuweg  
France

*FLX*

Citizenship

French

Post Office Address

same as above